Experimentelle Übungen zur Physik für Fortgeschrittene

Manual for the experiment

X-Ray-Reflectometry

written by

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1 Introduction

X-ray reflectivity (XRR) is a noncontact, non destructive technique to measure the near surface sturucture of materials. It probes the electron density contrast with depth resolution. As a consequence, it gives information on the surface or interface roughness, density and thickness of the sample. These samples could be single crystalline, polycrystallime or amorphous material.

As literature to this experiment we recommend the lecture of "Elements of modern x-ray physics" from Jens aks Nielsen [1] and in particular Chapter 3, pages 61-98 therein. Useful information about the analysis can be found in the book from Pietsch et.al., Chapter 8 [2].

2 Refraction of X-rays

The refraction index n of a medium for electromagnetic radiation depends on the frequency ω and thus the energy of the incoming wave. $n(\omega)$ shows resonates behavior if the energy of the incoming wave corresponds to an electronic transition within the atoms of the material. Before a resonant-frequency, n rises with increasing frequency (range of normal dispersion). Directly above the resonant frequency n drops strongly and rises up to the next resonant frequency again etc. The higher the frequency, the smaller becomes the value of the refraction index away from the resonant frequencies.
X-rays with energies around 10keV lie far above the binding energy of the most electrons of an atom. This has the consequence, that the refraction index for X-ray of normal materials is slightly **smaller** than 1. Usually one writes n in the form

$$n = 1 - \delta + i\beta \tag{1}$$

where the parameter δ considers the dispersion and β the absorption. These parameters are related to the linear absorption coefficient μ and the electron density ρ_e of the regarded material via:

$$\delta = \frac{2\pi}{k^2}\varrho_e r_0 \tag{2}$$

$$\beta = \frac{\mu}{2k} \tag{3}$$

$k = 2\pi/\lambda$ is the wavevector of the incoming wave with wavelength λ and r_0 the classical electron radius. Typically, δ is of the order 10^{-6} and β another order of magnitude below this. For the derivation of the formulas 2 and 3 we refer to the literature [2].

Snell's law, well-known from the optics, holds also for x-rays: During the transition from a medium with refraction index n_1 to a medium with refraction index n_2 the angles of incidence and exit, respectively, are related through:

$$n_1 \cos\alpha = n_2 \cos\alpha' \tag{4}$$

In x-ray physics, the angles of incidence α and exit α' are usually measured with respect to the

surface (see fig. 1).
For incident-angles below the critical angle $\alpha = \alpha_c$ it comes to total external reflection ($\alpha' = 0°$). If one inserts $n_1 = 1$ (air), $n_2 = 1 - \delta$ and $\alpha' = 0°$ into Snell's law and expands the cosine in a Taylor-series, then one receives the important relationship for the critical angle

$$\alpha_c = \sqrt{2\delta} = \frac{\sqrt{4\pi\varrho_e r_0}}{k} \tag{5}$$

For Cu-K_α-radiation and e.g. for silicon on obtains values of $\delta = 7,633 \cdot 10^{-6}$ and thus a critical angle of $\alpha_c \approx 0,23°$.

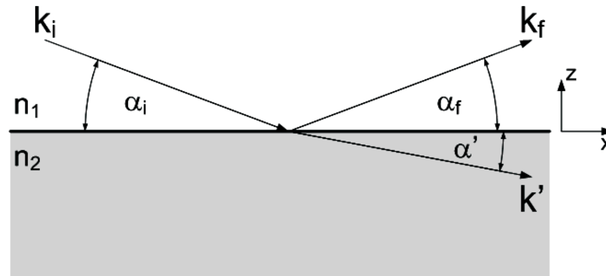


Figure 1: Reflection and refraction of x-rays

3 Fresnel-reflectivity of a smooth surface

For the determination of the reflectivity of a plane x-ray wave at an ideal smooth surface we can use the Fresnel-equations known from electrodynamics or optics. In our case, we don't need to differentiate between σ polarisation (electrical field-vector perpendicularly to the diffraction plane) and π polarisation (el. field vector parallel to the diffraction plane) due to the regarded small angles (in practice, reflectivity is usually examined up to an angle of incidence of about 5°). From the Fresnel-equations for σ polarisation we obtain the relations for the Fresnel reflection- and transmission coefficients for small angles:

$$r(\alpha) := \frac{E_r}{E_i} = \frac{\alpha - \alpha'}{\alpha + \alpha'} \tag{6}$$

$$t(\alpha) := \frac{E_t}{E_i} = \frac{2\alpha}{\alpha + \alpha'} \tag{7}$$

These equations refer to the *amplitudes* of the electrical field. One receives the appropriate coefficients R and T for the reflected and/or transmitted intensity through

$$R_F(\alpha) = \frac{I_r}{I_i} = |r|^2 \quad ; \quad T_F = \frac{I_t}{I_i} = |t|^2 \tag{8}$$

Here it is pointed out that the equations for the reflectivity always refer to so-called specular reflection, i.e. that for the reflected wave the condition angle of incidence = angle of exit must be fulfilled.

3.1 Penetration depth

From Snell's law for the refraction at a boundary between air and a medium with refraction index $n = 1 - \delta + i\beta$

$$\cos\alpha = n\cos\alpha'$$

Taylor expansion for small angles leads to

$$\begin{aligned} \alpha^2 &= \alpha'^2 + 2\delta - 2i\beta \\ &= \alpha'^2 + \alpha_c^2 - 2i\beta \end{aligned} \tag{9}$$

Thus equation 8 for the reflected intensity may be written as:

$$\mathrm{R}_F = \left| \frac{\alpha - \sqrt{\alpha^2 - \alpha_c^2 + 2i\beta}}{\alpha + \sqrt{\alpha^2 - \alpha_c^2 + 2i\beta}} \right|^2 \tag{10}$$

Figure 2 shows the Fresnel-reflectivity R_F as a function of the angle of incidence α for different values of β/δ. One recognizes that the reflectivity does not necessary have to be constant equal to 1 below the critical angle under any circumstances. Rather, a so called evanescent wave runs parallel to the surface inside the material. This phenomenon is already well-known from electrodynamics. The penetrating intensity is partly absorbed, whereby the reflectivity is reduced. The penetration depth λ of the evanescent wave, i.e. the depth, on which the penetrating intensity drops down to $1/e$ is given by

$$\Lambda = \frac{1}{2kIm(\alpha')} \tag{11}$$

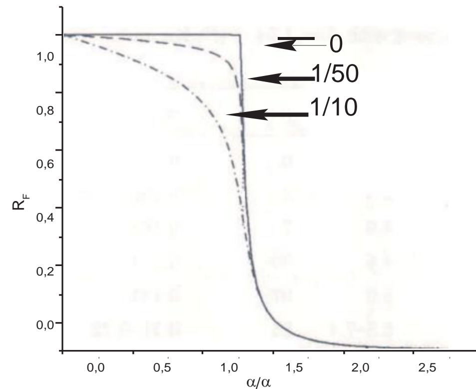


Figure 2: The Fresnel-reflectivity of a silicon vacuum surface ($\alpha_c = 0,\ 23°$) as a function of α_i/α_c for a wavelength $\lambda = 1,54$ Å. The different lines show different conditions of β/δ from 0 over 1/50 up to 1/10.

3.2 Reciprocal space

In the reciprocal space or impulse space, incident and reflected wave (incident angle, α_i, exit

angle of reflected wave α_f) are described with their wave vectors k_i and k_f, where $\dot{k} = 2\pi/\lambda$. In our geometry k_i,k_f and the surface-normal of the sample lie in a common plane, the refraction-plane. The impulse transfers are

$$Q_z = \frac{2\pi}{\lambda}(sin\alpha_i + sin\alpha_f) \qquad (12)$$

and

$$Q_x = \frac{2\pi}{\lambda}(cos\alpha_i - cos\alpha_f). \qquad (13)$$

where the surface-normal defines the z-direction and x lies in the refraction-plane parallel to the surface (fig.1). In this coordinate system, we have $Q_y = 0$. The vectors Q_z, Q_x and Q_y span the reciprocal space. In our case of specular reflectivity with $\alpha_i = \alpha_f := \alpha$, 13 becomes:

$$Q_z = 2k \sin\alpha \qquad (14)$$

The Fresnel's reflectivity is then determined as

$$R_F(Q_z) = \left| \frac{Q_z - \sqrt{Q_z^2 - Q_c^2 + 2i(2k)^2\beta}}{Q_z + \sqrt{Q_z^2 - Q_c^2 + 2i(2k)^2\beta}} \right|^2 \qquad (15)$$

The advantage of this method is the elimination of all setup specific factors of a measurement. For large angles of incidence, the Fresnel reflectivity can be replaced through

$$R_F(Q_z) \approx \left(2\frac{Q_z}{Q_c}\right)^{-4}$$

3.3 Surface-roughness

The previous considerations referred to homogeneous media with idealized, smooth surfaces. At realistic samples however, no abrupt change in the the density and/or refractive index takes place - the surface is always rough on atomic level (order of magnitude nm). This roughness causes an intensity reduction of the specular reflected wave and additional diffuse scattered intensity.

In order to determine the influence of these roughness on the reflectivity we examine the laterally averaged electron density

$$\rho_e(z) = \int\int \rho_e(x, y, z)dxdy \qquad (16)$$

The transition of a medium 1 with the electron density $\rho_{e,1}$ into a medium 2 with electron density $\rho_{e,2}$ can be described by a function $f(z)$:

$$\rho_e(z) = \rho_{e,1} + f(z) \cdot (\rho_{e,2} - \rho_{e,1}) \qquad (17)$$

For an ideally smooth surface, the change of electron density takes place abruptly, i.e. f(z) is just a step function. For a real, rough surface one usually uses a normalized gaussian distribution for the gradient of the electron density perpendicular to the surface:

$$\frac{\mathrm{d}f}{\mathrm{d}z} = \frac{1}{\sqrt{2\pi\sigma^2}} e^{-\frac{1}{2}\left(\frac{z}{\sigma}\right)^2} \qquad (18)$$

The parameter σ therein is the rms (root mean square) roughness, i.e. in the case of a sharp but however rough surface, σ^2 is the root-mean-square deviation of the surface-height $z(x, y)$ from its mean value (see fig. 3).

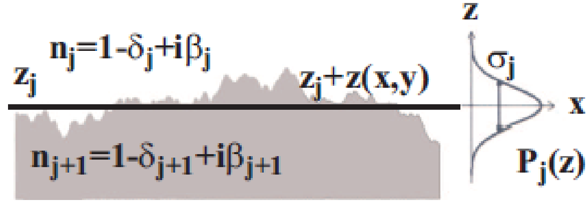


Figure 3: A rough surface with the mean height z_j has fluctuations $z(x, y)$ around this value.

The reflectivity of a rough surface differs from the ideal Fresnel-reflectivity only noticeable for angles above α_c. The ration from the reflectivity of the rough surface $R(Q)$ to the Fresnel-reflectivity $R_F(Q)$ is given by

$$\frac{R(Q)}{R_F(Q)} = \left| \int_0^\infty \left(\frac{\mathrm{d}f}{\mathrm{d}z} \right) e^{iQz} dz \right|^2 \tag{19}$$

With the model from Eq. 19, one obtains the important relation

$$R(Q) = R_F(Q) e^{-Q^2\sigma^2}. \tag{20}$$

4 Reflection from a thin slab

For the calculation of reflection at multi-layer systems, it is first useful to regard the reflection at only one thin layer with thickness Δ and refraction index n_1 on a substrate:

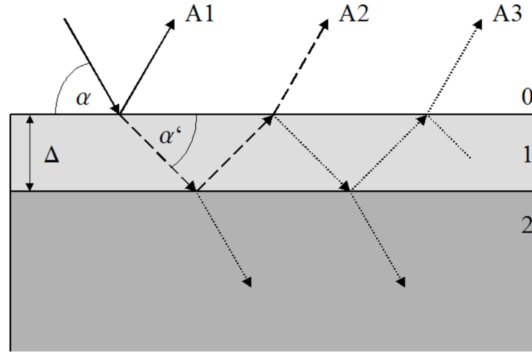


Figure 4: Reflection at a thin layer: The incident wave is reflected several times within the layer. The reflectivity arises as a result of correct summation of the individual reflected amplitudes.

The layer (1) sits on an "infinitely thick" substrate (2). Infinitely thick means that we can neglect reflections at the lower surface of the substrate. The reflection- and transmission coefficients for the transition air (0) - layer (r_{01}, t_{01}) and for the transition layer - substrate (r_{12}, t_{12}) are well known on the basis of the considerations in the previous section. The entire reflectivity r_{slab} is now the result from the summation of all waves reflected at the different boundaries (see fig. 4):

1. The wave with wave vector k, incident with the angle α, is first partly reflected at the surface: amplitude $A1 = r_{01}$
2. The wave that was transmitted with t_{01} is than partly reflected at the substrate and can escape again partly into air: amplitude $A2 = t_{01>}r_{12}t_{10}$. In relation to the first reflected

wave, this wave is now phase shifted due to the way through the layer and back about a factor $p^2 = e^{iQ_1 \cdot \Delta} = e^{i2k_1 \sin \alpha' \cdot \Delta}$.

3. After another set of reflections at the upper and lower surface of the layer, a wave with amplitude $A3 = t_{01} r_{10} r_{12}^2 t_{10}$ leaves into air. This is out of phase to the first reflected wave by a factor p^4.

and so on...

The total reflectivity of the thin layer can be obtained by correct summation of the individual amplitudes:

$$
\begin{aligned}
r_{Schicht} &= r_{01} + t_{01} r_{12} t_{10} p^2 + t_{01} r_{10} r_{12}^2 t_{10} p^4 + \ldots \\
&= r_{01} + t_{01} t_{10} r_{12} p^2 \sum_{m=0}^{\infty} (r_{10} r_{12} p^2)^m \\
&= r_{01} + t_{01} t_{10} r_{12} p^2 \frac{1}{1 - r_{10} r_{12} p^2}
\end{aligned} \tag{21}
$$

If we consider the conditions $r_{01} = -r_{10}$ and $r^2_0 + t_{01} t_{10} = 1$, following from the Fresnel-equations, the reflection coefficient of a thin layer can be written as

$$
r_{slab} = \frac{r_{01} + r_{12} p^2}{1 + r_{01} r_{12} p^2} \tag{22}
$$

with $p^2 = e^{iQ_1 \cdot \Delta} = e^{i2k_1 \sin \alpha' \cdot \Delta}$ and $k_1 = n_1 \cdot k$.

The reflection coefficient for the intensity $|r_{schicht}|^2$ shows the so-called "Kiessig oscillations" due to the phasse-factor p^2 with a period of $2\pi/\Delta$. Fig.5 shows the reflectivity of a thin layer of tungsten with a thickness $10 \cdot 2\pi$Å.

In the case of a substrate whose refraction index is smaller than that of the thin layer ($n_2 < n_1$), there are maxima within the reflectivity whenever $Q_1.\Delta$ is an integer multiple of 2π. With consideration of Snells law, we have the m'th maximum at an angle of incidence α_m given by

$$
\alpha_m^2 = \left(\frac{\lambda}{2\Delta}\right)^2 m^2 + \alpha_c^2 \tag{23}
$$

If we neglect the refraction effects ($\alpha_c \to 0$) this is the well-known Bragg-equation for small angles:

$$
m\lambda = 2\Delta \sin \alpha_m \tag{24}
$$

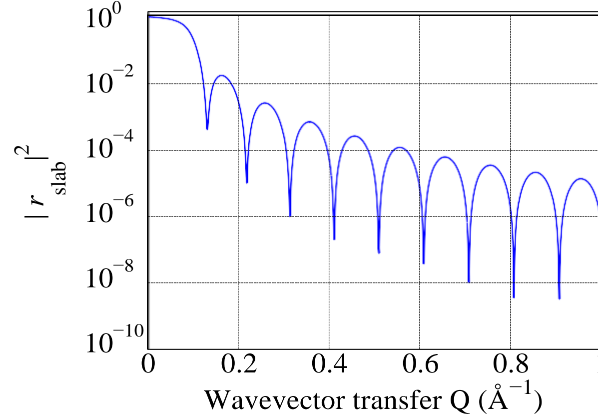


Figure 5: Kiessig-oscillations from a thin slab of tungsten

5 Reflection at multilayer systems - Parratts formalism

The result (22) can be extended to the case of a layer system consisting of N layers on a substrate by a iterative procedure. This formalism was invented 1954 by Parratt and therefore carries his names [6].
Let the layer system consist of N layers on an "infinitely thick" substrate. By definition, the N'th layer sits on the substrate and the zeroth layer is air and/or the surrounding medium. Each layer j has a refraction index $n_j = 1 - \delta_j + i\beta_h$ and the thickness Δ_j (fig. 6). From the solution of the Maxwell equations at the boundaries it follows that the x-component of the wavevector is conserved in all layers. For the wave vector in the layer j, the relation $k_j = n_j k$ holds and therefore, the z-component of the wavevector within the layer j is given by

$$k_{z,j}^2 = (h_j k)^2 - k_x^2$$

The wavevector transfer in the layer j is

$$Q_{z,j} = 2k_j \sin a_j = 2k_{z,j} \qquad (25)$$

If we neglect multiple reflections in a first step, the reflectivity at each interface between the layers j and $j+1$ can be calculated to (compare eq. (15)

$$r'_{j,j+1} = \frac{Q_{z,j} - Q_{z,j+1}}{Q_{z,j} + Q_{z,j+1}} \qquad (26)$$

Especially, the reflectivity between the lowest layer and the substrate can easy be calculated - here there is no multiple reflections - and we receive

$$r'_{N,\infty} = \frac{Q_{z,N} - Q_{z,\infty}}{Q_{z,N} + Q_{z,\infty}} \qquad (27)$$

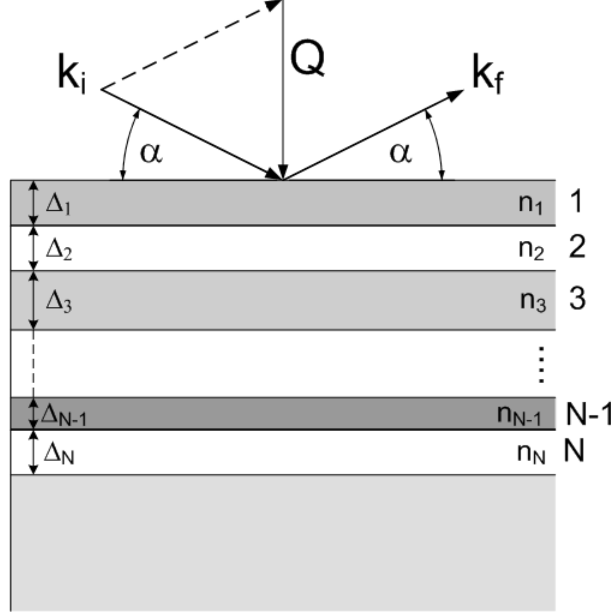


Figure 6: Parratts formalism: composition of the multilayer system

Now we treat multiple reflections: The reflectivity at the interface between layer N and N-1 is exactly the case of a thin slab on a substrate and can therefore be described with Eq.22 as

$$r_{N-1,N} = \frac{r'_{N-1,N} + r'_{N,\infty} p_N^2}{1 + r'_{N-1,N} r'_{N,\infty} p_N^2} \tag{28}$$

with the phase factor

$$p_j^2 = e^{iQ_{z,j}\Delta_j}.$$

With this reflectivity and Eq. 22 now again the reflectivity between layer N-2 and N-1 can be computed to be

$$r_{N-2,N-1} = \frac{r'_{N-2,N-1} + r_{N-1,N} p_{N-1}^2}{1 + r'_{N-2,N-1} r_{N-1,N} p_{N-1}^2} \tag{29}$$

and so on until we receive the reflectivity at the top of the multi-layer system.

The influence of roughness of the individual interfaces j on the specular reflected intensity can be taken into account by replacing the Fresnel coefficient by a new set of coefficients

$$\tilde{r}_{j+1,j} = r_{j+1,j} e^{-2k_{z,j}k_{z,j+1}\sigma_j^2} \tag{30}$$

where σ is the rms (root mean square) roughness of the interface j (compare fig.3).

6 Instrumental setup

Figure 7 shows the schematic setup of the diffractometer used for the reflection measurement. The detector and the x-ray tube lie on a circle and can be rotated around the common center, where the sample is placed. The detector is a scintillation counter/ photomultiplier with a monochromator-crystal placed in front. This serves for the fact that only the Cu-K_α-radiation can reach the detector. For small angles, especially for $\alpha_i < \alpha_c$ the reflected intensity is so high that a saturation of the detector arises. In order to avoid this, the first part of the reflectivity curve must be measured with a filter - simple a metal foil in suitable thickness - before the detector. For larger angles one needs no more filter and it is useful to increase the counting time per measured point to have better statistics.

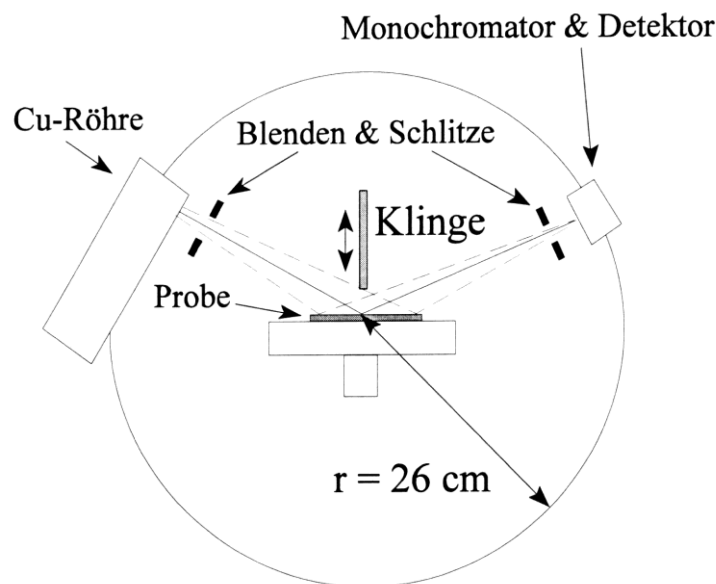


Figure 7: 2-Circle diffractometer

The X-ray beam used in the experiment is originated from Cu-Kα radiation and has a wavelength of 1.54A°. The sample used consists of a Si substrate, carbon (C) and tungsten (W) layers on top of it. At the anaysis part you should determine the number of layers with their thickness.

The sample must be aligned in such a way, that the condition angle of incidence = angle of exit is fulfilled. To achieve this, so called ω- scans must be performed. The sample is fixed in the middle of the diffractometer and the scattering-angle 2θ, that is the angle between primary beam and detector, is fixed to a constant value. However, it's unlikely that the surface normal the sample will be exactly vertical like in fig. 1 - there will be a certain angular deviation ω, so the angle of incidence and exit become $\alpha_i = \theta + \omega$ and $\alpha_f = \theta - \omega$, respectively. Because we cannot rotate the sample, we perform a scan where both x-ray tube and detector are rotated simultaneously in the same direction. The measured intensity will be clearly peaked when the specular reflection condition is met and the coordinate-system of our diffractometer can be rearranged so that the surface normal coincides with the z-direction like in fig. 1.

At the beginning of the experiment, you should check the alignment of the detector and the X-ray tube without the sample. Once you are sure that the alignment is good, then you should adjust the height of the sample. This can be done with the help of a metal blade inserted in between the x-ray tube and the detector such that the blade cuts the beam exactly in the middle. When this is done, the sample is inserted on the sample holder directly under the blade and then the blade is removed. Then the set up is ready for measurement.

You should have two different data sets. As explained above, for the smaller angles, you should use a filter and the counting time should be short, while for the higher angles, the filter would be removed and the counting time should be longer.

7 Evaluation of the measured curve

Since the measured curve consists of several parts, these must first be added together and normalized one on the other so that a continuous curve forms. The data has to be normalized in such a way, that the reflectivity at the critical angle - clearly visible in the curve - is unity. Theoretically one expects a constant reflectivity of 1 for angles of incidence below the critical angle. In practice, for small angles of incidence only a part of the incoming wave hits the sample - the so called "‘footprint"’ of the beam is larger than the sample. With increasing angle of incidence, a larger and larger fraction of the total beam falls on the sample; until finaly the entire beam falls on the sample and is reflected. Therefore one observes a sinusoidal rise of the measured reflectivity for angles below the critical angle (see fig. 8).
In order to correct this behavior caused by the experimental setup, one can calculate either this footprint effect accurately or simply set the reflectivity below the critical angle to the constant value 1. The ladder is sufficient for our purpose.

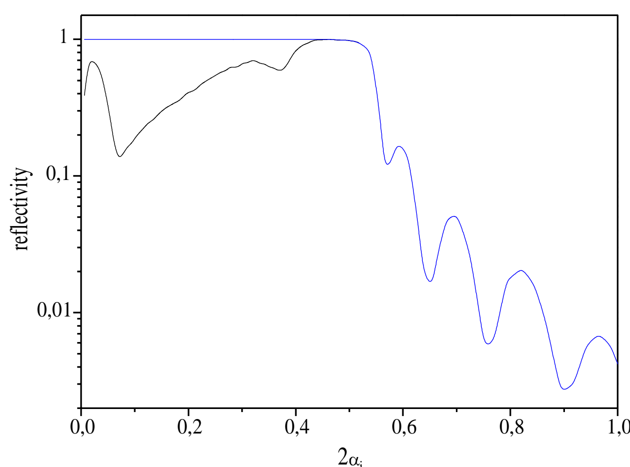


Figure 8: Reflectivity of a 20Ml leadstearate-film: measured (black) und corrected (blue).

7.1 Determination of layer thicknesses

By measuring the positions of the maxima on the refl. curve and the knowledge of equation 23 it is easy to determine the thicknesses of a double layer of stearate and also of the thickness of

the hole layer when it is sufficient large (approx. more than 6ML thick). When the peaks are very broad, it might be better to use the positions of the minima instead of the maxima, because they can be determined more accurate. In this case first calculate the equation for the positions of the minima (analogue to equation 23).

In addition to this, the position of the critical angles (if there is a substrate and a layer on top, there might be one critical angle for the layer and one for the substrate) can be determined directly from the reflectivity curve. These values are related to the electron densities of your sample, so you can calculate the densities of substrate and layer.

For further details and examples of this analysis from thin layers and multilayers see [7], Chapter 8 (attached to this manual).

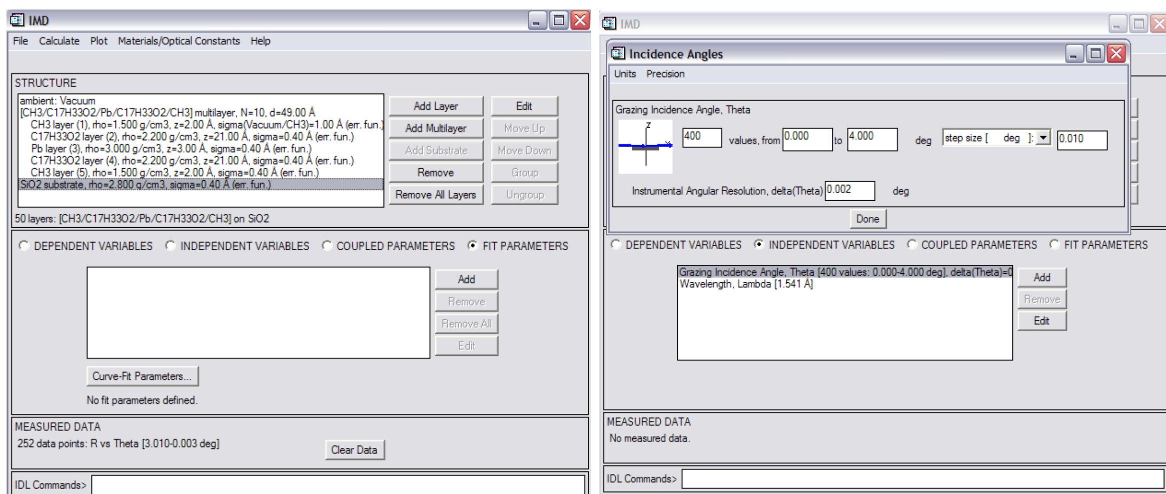
As a summary;

- you should plot the first data set and normalize the data and then combine this with the second data set keeping in mind that the overlaping sections of two data sets should have the same intensity value. You should draw the intensity scale of this graph (consisting of the two data sets) in *log-scale* to observe the patterns better.
- You should calculate the critical angle from this graph.
- The thickness, D, of the whole multilayer and the thickness, d, of a double layer can be calculated from the orders of the Kiessing fringes and the Bragg peaks respectively. You should determine the orders of the Kiessing fringes and the corresponding angles from the graph (you could use a Gaussian fit). You should plot and fit them linearly respectively. From the slope you could determine the thickness, while the interception point with the y axis will give you the critical angel (Eq.24).
- Once you have determined the D and d values, you should check if $D = 20d$.
- From the critical angle values you should determine the electron density. You should compare these values with the literature.

7.2 Simulation of the measured curve

The measured reflectivity should be simulated with the module "IMD" of the program "XOP" [1]. The program computes the reflectivity of an arbitrary layered system with Parratt's formalism described above.

For the simulation of the curves, first the data of the box model of the "ideal" structure must be feed into the program. Through "Add Multilayer" a periodic structure can be entered into the program. In the example from above, a double layer consists of five individual boxes. For each of these boxes either a material from a database implemented in the program can be selected or it's possible to enter the structure over the button "'Density and Composition"' by hand according to the chemical composition. The latter is recommended here. The density of the material is typically of the order 2,2g/cm^3 and is available later as a fit parameter. In addition, the thickness and the roughness can be entered here for each box. Illustration 13(a) shows the interface after input of a 20ML lead stearate film (10 repetitions à 5 boxes, here only roughly estimated input values).



7.2.1 Input of the 20ML model

(b) Input of the angular range

Figure 9: Program-interface

Under "Dependent variable" the quantity which should be computed has to be indicated, in our case the reflectivity. Under "Independent Var." the wavelength must be entered ($Cu - K_\alpha$) as well as the angular range for which the curve should be computed, e.g. 400 values between 0 and 4° with grazing incidence. Here also the instrumental resolution can be considered (fig. 9 (b)).

Through the tab "Calculate - > Specular..." the reflectivity curve of the model can be computed (- > fig. 10).

[1] A three-box modell where you combine the CH3 Group with the long tail might be sufficient or our purpose and is much easier to handel, because the number of fit-parameters is reduced. We recommend that you use such a three-box model.

Figure 10: Simulated reflectivity curve of the above model

In order to import the data into the program, these must be present in a ASCII file (ending .dat) with two or three columns - 1.column: α_i or Q_z, 2.column: R (normalized on 1), optionally 3. Column: σ_r. In order to reduce calculation time, it's possible to shrink the data set with possibly far over 1000 measured points something, for example by skipping every second point. It is also good to make sure that the used data are still useful at all; especially for high angles the counting rate decreases strongly. The import of the data can be done over "File -> open Measured...".

Under the point "coupled parameter" in the IMD window, parameters can be linked with one another. For example, it is reasonable that the two CH3-boxes or the tails in our example have got the same density, thickness etc.. In order to fit the data, under "‘fit parameters"’ a large number of different fit-parameters can be selected. Initial values and upper/ lower constraints can be indicated in each case. The parameter R_{scale} can be carried within the fitting, but however should lie near unity in the end. To fit the data, it's not a good idea to fit all possible parameters at once. It's better to do it one after the other, for example starting with the layer thicknesses, then perhaps the densities etc.. Unfortunately the program does not take over the final values after the fit as new initial value. This has to be done by hand. In addition one should not trust the program blindly, but always check whether the respective parameter value is still meaningful at all. With a system with so many parameters the algorithm easily runs into a local minimum and stucks. The fit procedure is quite complex and lengthy therefore with such a system. The fit-algorithm can be adjusted under "curve-fit of parameter" to logarithmic fit (without priority, otherwise one gets error messages).

Requirements for the Report:
The report should cover what is done during the experiment, the analysis of the data and what is learnt through the process. It should contain the below sections:

- **Introduction:** What is the lab course about? What is the aim of the experiment? What have you learned?
- **Theory:** The background information for this experiment. (Be sure that you include the following topics; How does density, thickness, roughness, etc. affect your measurement? Information provided by this experiment)
- **Experimental:** Description of setup and procedure.
- **Result:** Exhibit the experimental results and your analysis.
- **Conclusion**
- **References**

References

[1] Als-Nielsen, Jens; McMorrow, Des, Elelments of Modern X-ray Physics, John Wiley and Sons, 2001

[2] Pietsch, Ullrich; Holy Vaclav; Baumbach, Tilo, High-Resolution X-ray Scatttering: From Thin Fims to Lateral Nanostructures, Springer, 2004