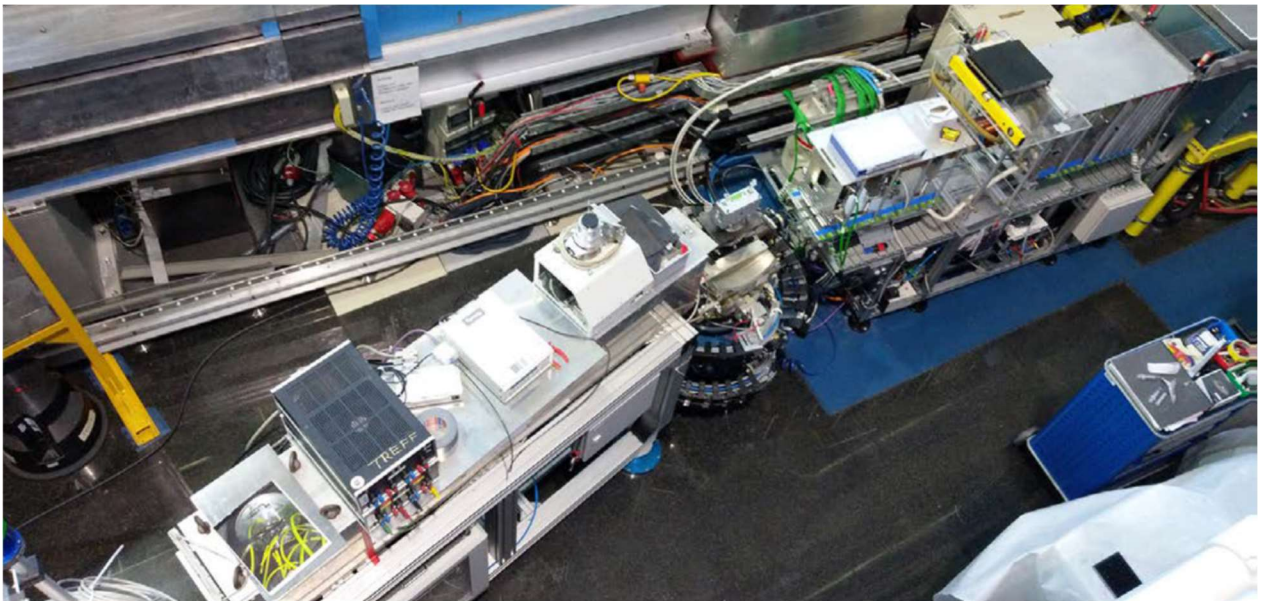


# Advanced TREFF-lab course for Master students: Neutron reflectometry

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December 2021



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# 1 Introduction and Experimental purpose

Neutron Scattering (NS) is a well-established and dedicated method to analyze structures and dynamics in all areas of condensed matter research. Accordingly, the field of application of NS covers a wide range of fields such as the determination of structure properties from the microscopic to mesoscopic scale in atomic, molecular or magnetic systems, the study of real material structure and texture including residual stresses, as well as the determination of lattice vibrations, transport processes and magnetic excitations, etc.

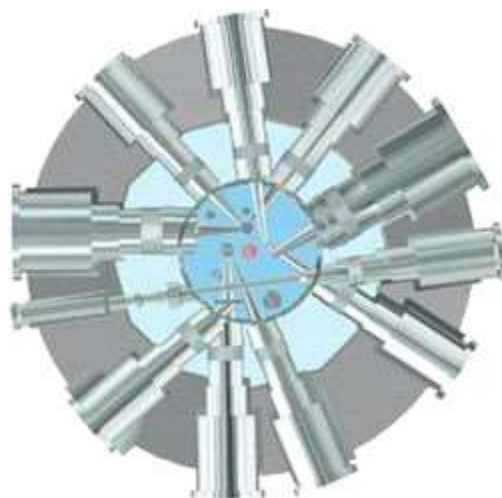
Neutron reflectometry is also excellent for probing detailed surface structures as well as boundary surface structure- and thin-film systems.

In this experiment, carried out at the neutron reflectometer TREFF located at the FRM II (Forschungsreaktor München II), various properties of neutron supermirrors will be examined as a hand-on example of a layer system and the basics of the reflectometry technique will be learned.

As a key component of neutron guides, neutron supermirrors have a major impact on the quality of instruments installed at the FRM II.

## 2 The FRM II

The **Research Neutron Source Heinz Maier-Leibnitz** (FRM II) is a beam tube reactor optimized for providing a very high thermal neutron flux. The concept is primarily based on the use of a compact core containing a single cylindrical fuel element installed in the center of a moderator tank filled with heavy water ( $D_2O$ ). The cooling is carried out with light water from the reactor pool. The reactor itself is controlled by means of the central control rod inside the fuel element. To ensure a reliable „switching off“, an independent shut-down system consisting of five shutdown rods in the moderator tank has been realized. Every single one of these shutdown rods is capable of instantly and permanently turning off the reactor at any given time.



*Figure 1: Horizontal section of the reactor pool filled with heavy water (simplified schematic) 300mm above core center level. The fuel element is located in the center. The beamlines are arranged tangentially to the core.*

The compact design of the fuel element and the heavy water  $D_2O$  acting as a neutron moderator require that the maximum thermal neutron flux density (usually around

$8 \times 10^{14} \text{ cm}^{-2}\text{s}^{-1}$ ) is located at a distance of about 12 centimeters of the fuel element inside of the moderator tank. This section of maximum flux density is primarily used for experiments. Right here, distributed evenly around the circumference, a number of beam tube noses and various vertical irradiation channels are arranged.

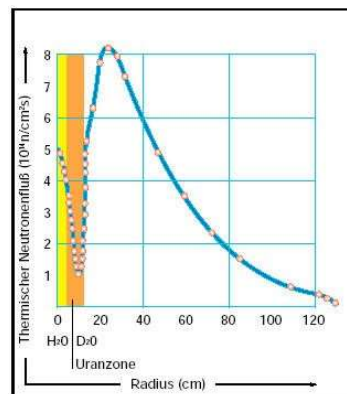


Figure 2: Thermal neutron flux density as a function of the radius at half of the fuel element's height, (when unaffected, i.e. without any obstacles in the tank). The maximum flux is approximately 12 cm outside of the fuel element inside of the moderator tank.

The current experimental facilities and laboratories are located outside of the reactor pool as an extension of the beam lines inside of the experimental hall and along the neutron guides in the neutron-guide hall. The neutron-guide hall connects the FRM-II-building with its predecessor, the FRM I, also called the „Atomic-Egg“. The neutron-guide hall provides additional space for experimental uses almost the size of the experimental hall. Additional information on the FRM II, its instruments and scientific used can found in the booklets<sup>1,2</sup>.

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<sup>1</sup> [https://mlz-garching.de/index.php?article\\_id=91&file=experimental-facilities\\_online.pdf](https://mlz-garching.de/index.php?article_id=91&file=experimental-facilities_online.pdf)

<sup>2</sup> [https://mlz-garching.de/files/20170601\\_neutrons-for-research\\_online.pdf](https://mlz-garching.de/files/20170601_neutrons-for-research_online.pdf)

## 3 Theory

### 3.1 Neutron diffraction in general

In the following section we will determine some of the fundamental properties of neutron waves, such as the refractive index of matter for neutrons or the critical angle of total reflection and their relation. In terms of waves, the De-Broglie equation is applicable:

$$k = \frac{2\pi}{\lambda}, p = \hbar k \Rightarrow E = \frac{\hbar^2 k^2}{2m}$$

Neutron optical phenomena are generally based on coherent elastic scattering. The characteristic key feature of this scattering is the fact that it does not cause changes in state and properties of microscopic particles. Thus, it can be described using a time independent Schrödinger equation for a free particle:

$$\left\{ -\frac{\hbar^2}{2m} \Delta + V(\vec{r}) \right\} \psi(\vec{r}) = E \psi(\vec{r}) \quad (1)$$

Where  $V(r)$  denotes the optical potential, describing the effective interaction between the neutrons and the system,  $E$  stands for the Energy and  $\Psi(r)$  for the wave function of the incoming neutron.

The interaction of a neutron with an atom is composed of the strong interaction with the nucleus, as well as the electromagnetic interaction with the electron shell. The most important contribution, however, is the strong interaction, whose potential has an effective range of the order of the nuclear radius ( $R \approx 1$  fm).

It is well known from general scattering theory that the scattering of a wave with a given wavenumber  $k$  by a potential of an effective range  $R$  is isotropic, as long as  $kR \ll 1$  (s-wave scattering). For thermal neutrons, we may approximate:

$$k \approx 1\text{\AA}^{-1} \Rightarrow kR \approx 10^{-5} \ll 1$$

Therefore, this condition is best fulfilled and the wave leaving the scattering center is spherically symmetric. The core potential can be understood as a point interaction (Fermi pseudopotential):

$$\begin{aligned} \Psi_{scat} &\propto \frac{-b}{r} e^{ikr} && \text{(Spherical wave)} \\ V(\vec{r}) &= \frac{2\pi\hbar^2}{m} \delta(r)b && \text{(Fermi - Pseudopotential)} \end{aligned} \quad (2)$$

where b is the so called coherent scattering length, which can be determined experimentally. For homogeneous materials it is defined as:

$$V_0 = \frac{2\pi\hbar^2}{m} Nb$$

where N describes the nuclear density.

With this assumption, it is also possible to calculate the total effective cross section for scattering in one localized scattering core:

$$\begin{aligned} \Phi &= \text{neutron flux in } \frac{1}{\text{cm}^2\text{s}} \text{ units} \\ \sigma &= \frac{\text{Total number of scattered neutrons per second}}{\Phi} \\ \frac{d\sigma}{d\Omega} &= \frac{\text{Total number of scattered neutrons per second emitted into the solid angle element } d\Omega}{\Phi d\Omega} \end{aligned}$$

Being v the velocity of a neutron (before and after scattering), the neutron flux per second over area element dS after scattering is:

$$vdS |\Psi_{scat}|^2 = vdS \frac{b^2}{r^2} = vb^2 d\Omega$$

The incident flux of a plane wave is:

$$\begin{aligned} \Phi &= v |\Psi_{incident}|^2 = v \cdot 1 \frac{1}{\text{cm}^3} \\ \frac{d\sigma}{d\Omega} &= \frac{vb^2 d\Omega}{\Phi d\Omega} = b^2 \Rightarrow \sigma_{tot} = 4\pi b^2 \end{aligned} \quad (3)$$

The scattering center (a nucleus) is here assumed to be immobile, therefore no kinetic energy can be transferred. Moreover, the neutron energy is too small to excite

possible resonances of the scattering nucleus (elastic scattering). In case of scattering on multiple nuclei (core position  $\vec{R}_i$ ) one obtains:

$$\Psi_{incident} \propto e^{i\vec{k}_0\vec{R}_i} \text{ incoming wave at the core } i$$

$$\Psi_{scat} = \sum_i e^{i\vec{k}_0\vec{R}_i} \frac{-b_i}{|\vec{r} - \vec{R}_i|} e^{i\vec{k}'(\vec{r} - \vec{R}_i)}$$

$$\frac{d\sigma}{d\Omega} = \frac{v dS |\Psi_{scat}|^2}{v d\Omega} = \frac{dS}{d\Omega} e^{i\vec{k}'\vec{R}} \left[ \sum_i e^{i\vec{k}_0\vec{R}_i} \frac{-b_i}{|\vec{r} - \vec{R}_i|} e^{i(\vec{k}_0 - \vec{k}')\vec{R}_i} \right]^2$$

If the measurement takes place far away ( $r \gg R_i$ ), one can approximate  $d\Omega = dS/r^2$ .

Summing over the scattered wave functions one gets:

$$\frac{d\sigma}{d\Omega} = \sum_{i,j} b_i b_j e^{i(\vec{k}_0 - \vec{k}')(\vec{R}_i - \vec{R}_j)} = \sum_{i,j} b_i b_j e^{i\vec{Q}(\vec{R}_i - \vec{R}_j)} \quad (4)$$

$\vec{Q}$  is here the momentum transfer  $\vec{Q} = \vec{k}_0 - \vec{k}'$ . The scattering length  $b_i$  is specific for each isotope. It depends on the spin relative to the incident neutron and the eigenstates of the nucleus. For a single nucleus  $b_i = \langle b \rangle + \delta b_i$  applies, where  $\delta b_i$  averages to 0.  $b_i b_j = \langle b \rangle + (\delta b_i + \delta b_j) + \delta b_i \delta b_j$ , but  $\langle \delta b \rangle = 0$  and  $\langle \delta b_i \delta b_j \rangle = 0$  except for  $i=j$ . Thus,  $\langle \delta b_i^2 \rangle = \langle b_i - \langle b \rangle \rangle^2 = \langle b^2 \rangle - \langle b \rangle^2$ . It is then:

$$\frac{d\sigma}{d\Omega} = \overbrace{\langle b \rangle^2 \sum_{i,j} e^{i\vec{Q}(\vec{R}_i - \vec{R}_j)}}^{\text{kohärent}} + \overbrace{(\langle b^2 \rangle - \langle b \rangle^2) N}_{\text{inkohärent}} \quad (5)$$

The first term contains a phase information of the scattered waves ( $\Rightarrow$  coherent component). It depends on the momentum transfer's  $\vec{Q}$  direction, i.e. it measures the relative position of the atoms relative to each other.

The incoherent component, on the other hand, is isotropic; it occurs as a background in the elastic measurement and is comparable to the Debye-Waller factor known from solid-state Physics.



Question 1: What results for the coherent term, when we consider the wave vector change  $\vec{Q}$  in the reciprocal space?

### 3.2 Refractive index and total reflection

Now we are able to determine certain material properties, which are relevant for neutron optics purposes, such as the refractive index or the critical angle for total reflexion. The general solution of Schrödinger's famous equation is a superposition of plain waves:

$$\Psi(\vec{r}) = \begin{cases} \sum A e^{i\vec{k}'\vec{r}} & \text{in a medium} \\ \sum A e^{i\vec{k}\vec{r}} & \text{in a vacuum} \end{cases}$$

$$\Rightarrow E = \frac{(\hbar k')^2}{2m} + V_0 = \frac{(\hbar k)^2}{2m}$$

Snellius' Law also applies for neutrons:

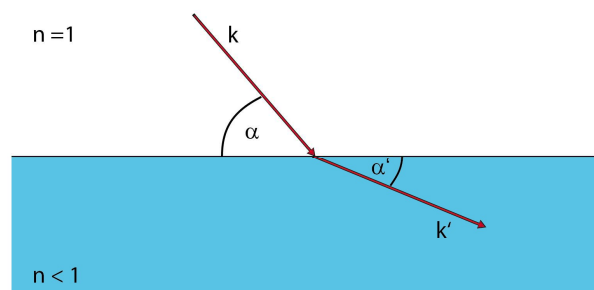


Figure 3: Refraction - neutron beam along  $k$

This gives for the refraction (analogous to classical light optics)

$$k \cos \alpha = k' \cos \alpha' \Rightarrow \cos \alpha = n \cos \alpha' \quad (6)$$

Where the refractive index  $n$  is defined as:

$$n^2 = \frac{k'^2}{k^2} \Rightarrow n^2 = 1 - \frac{V_0}{E} \quad (7)$$

and thus can also be written as:

$$n = \sqrt{1 - \frac{4\pi\rho b}{k^2}} = \sqrt{1 - \frac{\lambda^2}{N_b}} \quad (8)$$

If the following condition applies for equation (6):

$$\alpha' = 0 \Rightarrow \cos \alpha_c = n$$

total reflection occurs for incident angles smaller than the critical angle  $\alpha_c$  (total external reflection instead of total internal reflection in classical optics).

The value for the refraction index is (see table 1):  $n \approx 1$  i.e. one can expand cosine and thus the following relationship holds for the critical angle:

$$\begin{aligned} \cos \alpha_c &\approx 1 - \frac{\alpha_c^2}{2} + \dots = n \\ \Rightarrow \alpha_c &= \sqrt{2 - 2n} = \sqrt{\frac{N_b}{\pi}} \lambda \end{aligned}$$

This certain critical angle can now be translated into a critical transfer of wavevector  $q_c$ , which marks the beginning of total reflexion (see Figure 4). The transfer of wave vector perpendicular to the surface is given by:

$$q = \frac{4\pi}{\lambda} \sin \alpha$$

Thus, when the critical angle is used,  $q_c$  applies:

$$q_c = 4 \sqrt{\pi N_b}, \text{ bzw. } q_c = \frac{4\pi\alpha_c}{\lambda}$$

You can find the values for  $q_c$  of selected materials in table 1 of the appendix.

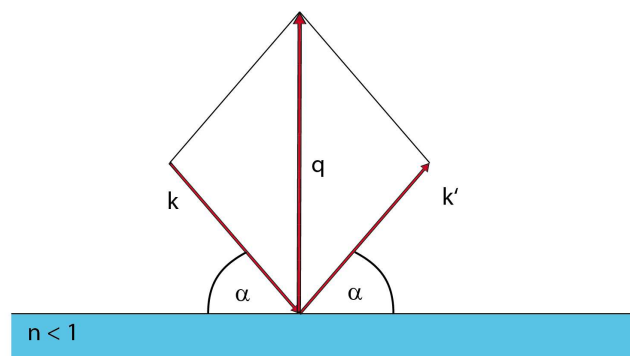


Figure 4: transferred wave vector  $q$  perpendicular to the surface - neutron beam along  $k$ .

For a layered system, e.g. a layer of Ni on a glass substrate, analogous considerations can be made. It turns out that similar to optics in multilayer systems, minima and maxima occur in the reflection due to the interferences of the waves reflected back from different interfaces. In the simple case of a single nickel layer of thickness  $d$  on a glass substrate, the periodicity of these regular, so-called Kiessig oscillations depends on the layer thickness  $d$  as given in (9) (see also Fig. 5).

$$Q = 2\pi/d \quad (9)$$

Not taken into account in these considerations are potential influences such as:

- \* surface roughness,
- \* resolution of the measuring facility,
- \* real velocity distribution of neutrons,
- \* boundary interfaces within sample
- \* and contamination of the sample.

which makes that a calculation adapted to reality can become very complex. Various software packages exist for the evaluation of measurement data of more complex layer systems.

In addition to this simplified thickness determination of the nickel layer, there is a more exact method based on the application of the Bragg relationship.

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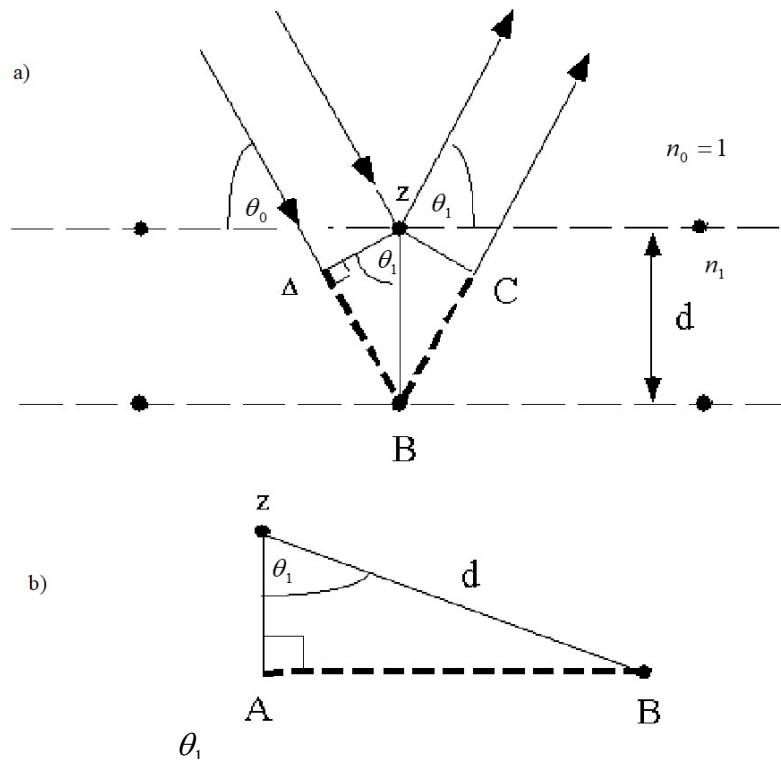


Figure 5: reflection on a thin layer of thickness  $d$ : a) shows the incident and the reflected wave's geometry b) shows the triangle  $ABZ$  in detail, which is being used for the calculation of the difference in path lengths of the beam path.

The Bragg condition states, that constructive interference only happens if the length difference of the optical paths is an integer multiple of the wavelength:  $\Delta = 2d \sin \theta_1 \cong 2d\theta_1 = m\lambda$  (Figure 6),  $d$  being the layer thickness in the crystal,  $\lambda$  being the wavelength of the incident neutron beam ( $4.74\text{\AA}$  in our case) and  $m$  an integer.

Futhermore the optical law of refraction is used:

$$\left. \begin{array}{l} n_0 \cos \theta_0 = n_1 \cos \theta_1 \\ n_0 = 1 \\ n_1 = 1 - \delta + i\beta \\ \cos x = 1 - \frac{x^2}{2} \end{array} \right\} \Rightarrow \theta_1 = \sqrt{\theta_0^2 - 2\delta + 2i\beta}$$

Absorption of the neutrons can be neglected:

$$m\lambda = 2d\sqrt{\theta_0^2 - 2\delta} = 2d\sqrt{\theta_0^2 - \theta_c^2}, \text{ with the use of } \theta_c = \sqrt{2\delta}.$$

And finally

$$\theta_m^2 = \left(\frac{\lambda}{2d}\right)^2 m^2 + \theta_c^2 \quad (10)$$

where  $\theta_m$  describes Bragg peak of the n-th order. If one plot  $\theta_m^2$  (in radians) versus  $m^2$ , a linear behavior is. By using a lineal fit one can get information about the layer thickness (encoded in the slope of the fitting) and the critical angle of total reflection  $\theta_c$  (encoded in the Y – axis intercept.). Once  $\theta_c$  is known, one can calculate the critical wave vector  $q_c$  and thus, the material can be identified.

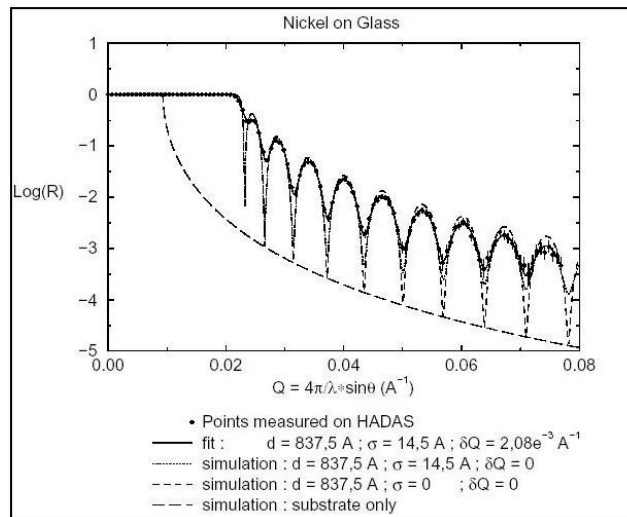


Figure 6: Reflectivity curve of a Ni layer on a glass substrate, simulated measurement curves show the contributions of substrate and surface roughness.

Figure 7 illustrates the use of nickel or nickel / titanium layer systems in neutron optics. A neutron guide made of mirrors with a single nickel layer transports neutrons up to the critical angle of total reflection ( $\sim 0.1^\circ/\text{\AA}$ ) without loss. Mirrors with multiple layers of e.g. Ni and Ti have an additional reflection maximum whose angle depends on the layer thickness  $d$ . By varying the layer thickness over many pairs of layers, one can

produce so-called supermirrors whose reflectivity remains close to 1 far beyond the critical angle of total internal reflection. These super mirrors are characterized by the m-value, which indicates by which factor the truncation angle of the reflectivity curve exceeds the critical angle of total reflection of Ni.

Question 2: What is the m-value of the supermirror displayed in figure 7 (right)?

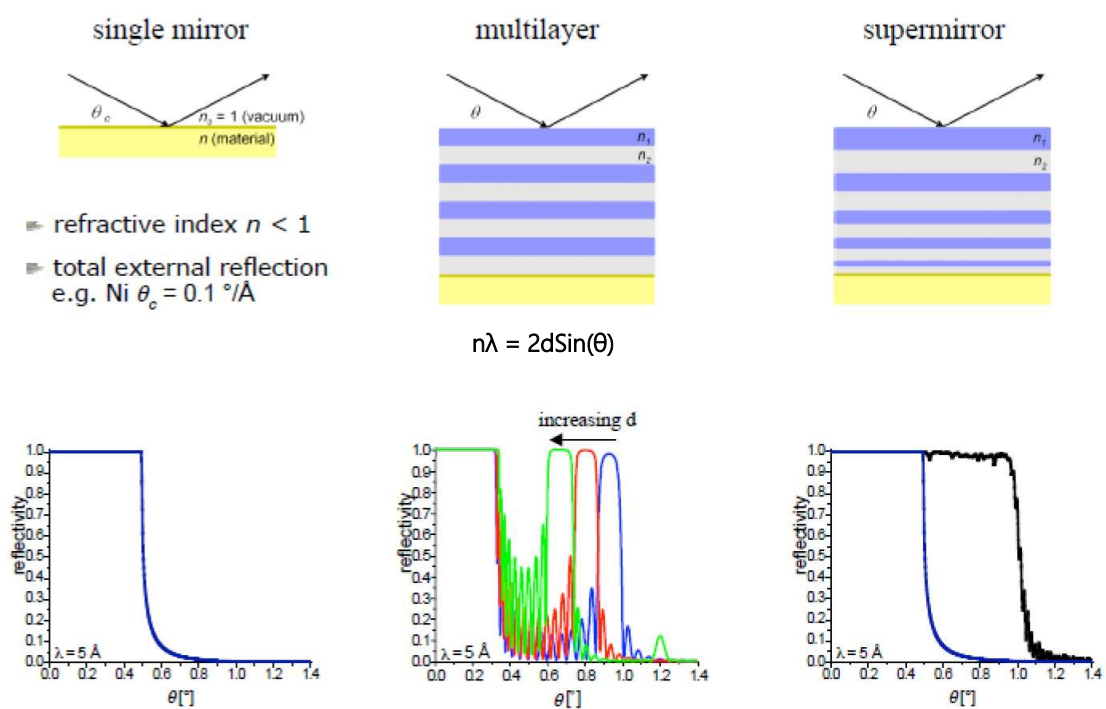


Figure 7: full range of layer systems from simple single mirror to super mirrors

## 4 Experimental procedure

The aim of this experiment is to investigate 2 to 3 samples of different layer systems on glass substrates made out either of Ni or Ni/Ti. Students have to: 1) calculate the layer thickness of the samples by using measurement data and 2) make an assessment of the coating sample examined. At the beginning of the course, the supervisor will instruct briefly on the safety systems and operation of the instrument.

As a legend for the following experiment description: Device names, *omega* (rotation of the sample) as well as commands which can be entered on the command line, e.g. *count(2)*, are printed in *italics*. The supervisor will give you a short introduction to the most important features of the software used in all FRM II facilities for controlling instruments, named NICOS.

### 4.1 Instrument configuration

Figure 8 illustrates the coordinate system at TREFF's sample position. There you can also find the possibility to adjust the different angles and axes of the instrument.

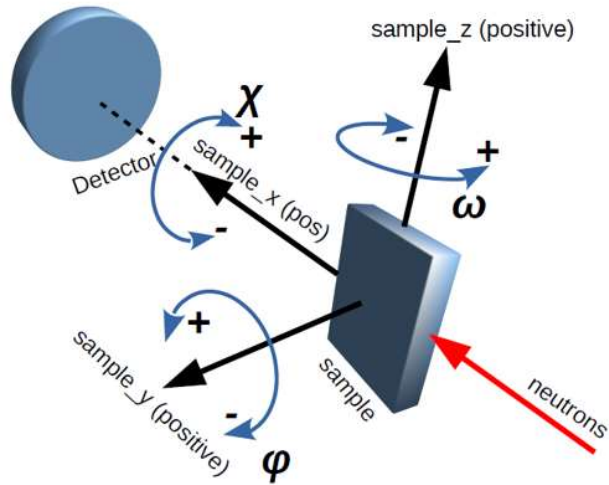


Figure 8: Coordinate system and controllable axes of TREFF's sample position

## 4.2 Primary Beam Definition

The two slit apertures (slit *s1* and *s2*) in the primary collimation path allow to define a narrow parallel beam. When the detector arm (motor *detarm*) is at an angle of  $2\Theta=0^\circ$ , the beam hits the central point of the detector after having passed through the vertical rotation axis ( $\Theta$ ) of the sample table (motor *omega*). The distance between the two apertures is about 1500 mm. Both apertures can be adjusted in both width and height with the following command:

$$\text{move}(sx,[0.4,30]), x=1,2$$

In this example, the change in width would be 0.4 mm and in height 30 mm.

The next step to perform is the adjustment of the zero point of the detector arm angle. For determining the zero direction of the neutron beam the instrument's detector arm angle  $2\Theta$  (motor *detarm*) has to be scanned around 0 degrees with 0.1 degrees interval. 15 positions in the left side and another 15 in the right side will be scanned, with a measurement time of 2 seconds per point. The command to do so



would be:

*cscan(detarm, 0, 0.1, 15, preset=2)*

The result of this calibration should look similar as shown in Figure 9.

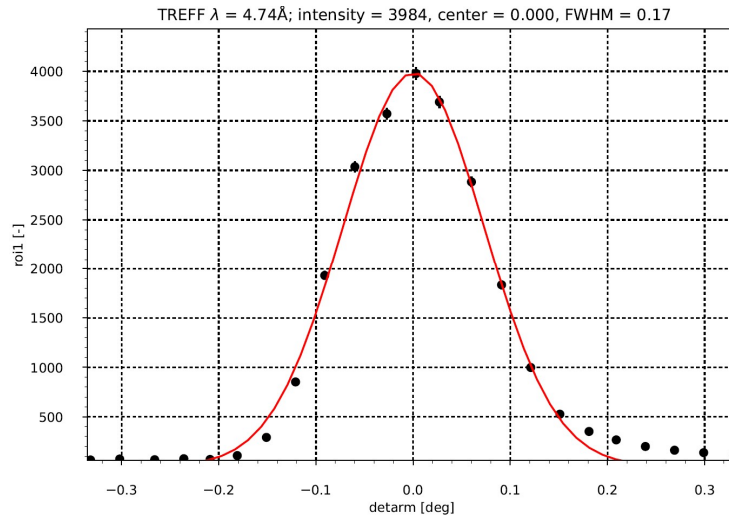


Figure 9: Testing of the detector angle adjustment with the direct beam.

The measured peak allows a direct determination of the primary beam's direction. By using the commands:

*move(detarm, Peak center)* and *adjust(detarm,0)*

the detector arm is moved to the position with the highest beam intensity and a new zero point is defined if necessary.

### 4.3 Sample alignment

Now the sample is ready to be fixed on the sample holder. A laser beam is working as an auxiliary device, which allows for a coarse pre adjustment of the sample's height and direction. That way the experimenter can be sure that its surface is aligned parallel to the primary beam, at least to first order. In order to achieve the parallelism it might be necessary to move the axes for sample rotation (motor *omega~0*) and sample tilt

(motor  $\chi=0$ ) in small steps until the laser beam is being reflected back onto itself from the mirror surface.

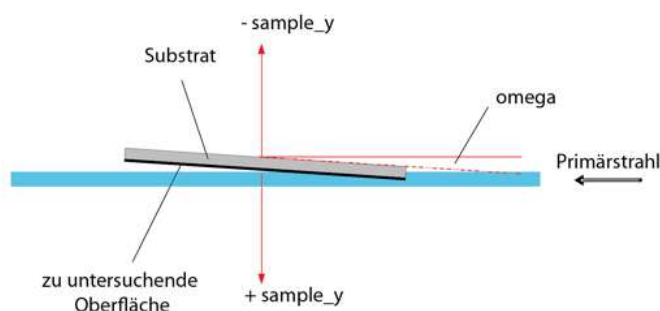


Figure 10: position of the sample relative to the primary beam. By adjusting  $\omega$  and  $sample\_y$  the sample surface can be aligned centrally, in parallel to the primary beam.

Next step is to position the middle of the sample in the center of the neutron beam. The aim is to align the sample surface exactly centrally, in parallel to the primary beam (see Figure 10). This requires both the axes for sample translation  $sample\_y$  and sample rotation  $\omega$  to be scanned. By entering the command:

*$sscan(sample\_y, start\ point, step\ size, number\ of\ points, measuring\ time)$* ,

the sample position is scanned, whereby initial value and step interval are both specified in mm. The result of the scan should look similar to Figure 11.

After having successfully moved the sample to the center of the beam, now the probe has to be aligned exactly parallel to the beam direction. The substrate under the sample is absorbing and thus, a scan of the sample rotation  $\omega$  results in a maximum intensity at the detector exactly when the sample is parallel to the primary beam (cf. Figure. 10):

*$cscan(\omega, 0, 0.05, 10, preset=2)$*

Just as before with the detector arm, now the zero point of the sample rotation angle

has also to be calibrated to zero degrees.

*move(omega, peak center) ; adjust(omega,0)*

After that, the *sample\_y* scan is done once again, to further refine the position, so that the sample position is set to the value where the beam has half intensity (see Figure 11). Now the sample is parallel with the beam and adjusted to be half way in the beam.

Explain the reason of this repetition using Figure 9.

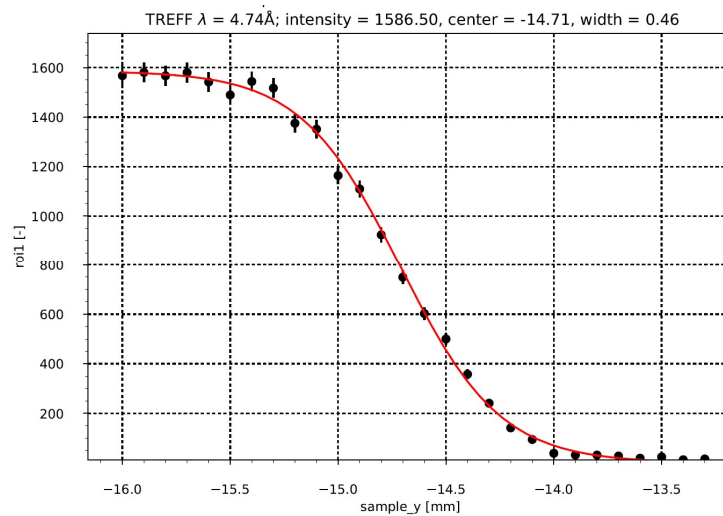


Figure 11: characteristic and typical scan of the sample position *sample\_y*.

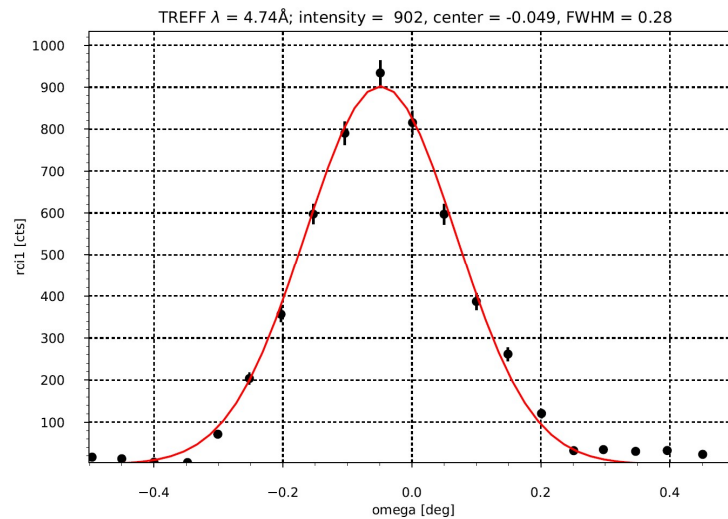


Figure 12: characteristic scan of the sample rotation angle *omega*.

The TREFF instrument is equipped with a 2D detector, which means that the beam

intensity distribution in the detector can be spatially checked and resolved. The actual measure is the integrated count rate in the area inside the red frame. (Region Of Interest, *roi*). In order to control the sample alignment, the centering of the scattered beam in the detector can be checked. To do so, the sample has to be arranged in reflection-position at low angles (at which total reflection is expected) and then a short counting can be performed with the command *count(2)*.

Calculate the critical angle of total reflection for Ni and glass (SiO<sub>2</sub>) using the data from Table 1 for the only available wavelength of  $\lambda = 4.74 \text{ \AA}$  at the reflectometer TREFF.

When the sample alignment is correctly performed, the result will be a detector image similar to Figure 13, including the scattered beam intensity within the red frame of *roi*.

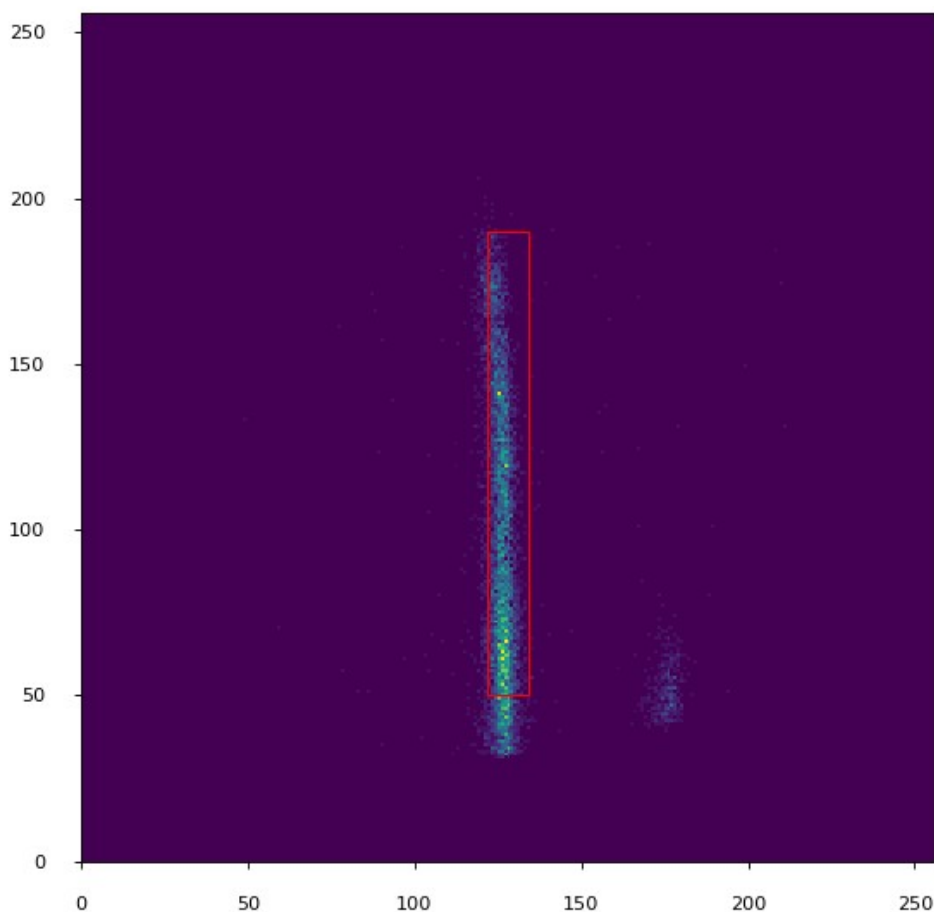


Figure 13: detector image at reflection condition  $\theta$ - $2\theta$ .

## 4.4 Measurement

For the measurement of the reflectivity curve itself, you use the coordinated movement of  $\theta$  and  $2\theta$ , i.e. of the device axes *omega* and *detarm*, by using the logical axes *t2t* for the scan, where Start Point and Step Size both refer to the angle  $\theta$ .

*sscan(t2t, Start Point, Step size, number of points, measuring time)*

Due to the fact that the measured beam intensity decreases very rapidly at larger scattering angles, please consider to make at least 2 or 3 partial scans with different count times. Take the counting rate at total reflection as a reference for orientation.

The result should look similar to Figure 14. The whole measurement procedure including the sample alignment and the measurement scan has to be done for each individual sample separately.

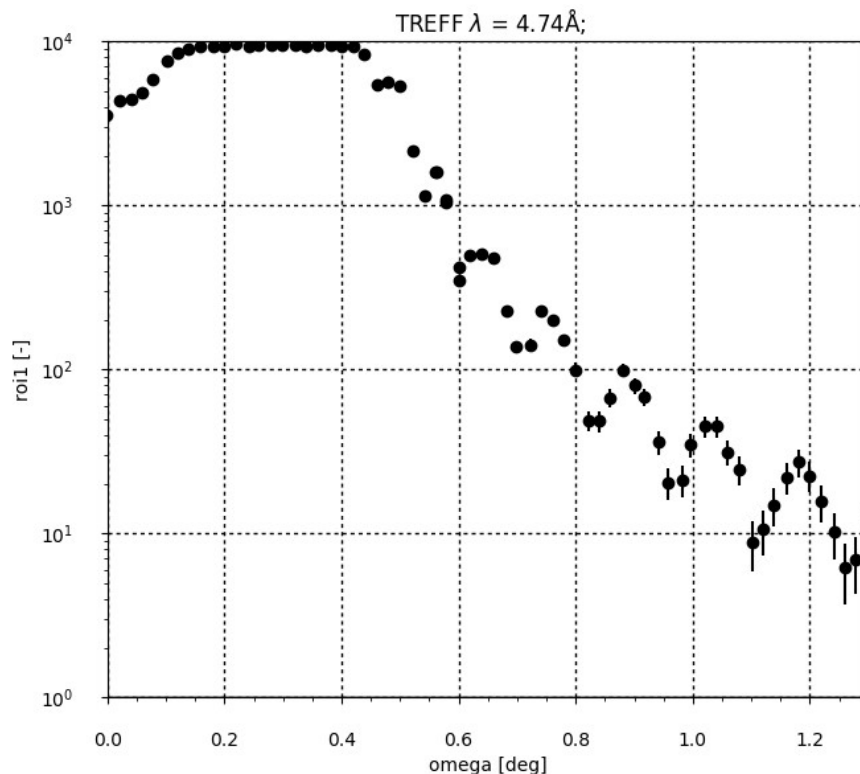


Figure 14: raw data in logarithmic scale of a  $\theta$ - $2\theta$  scan vs.  $\theta$  (*omega*). Note – this data is composed of 2 partial scans with different counting times.

## 5 Data evaluation

Convert your raw measurement data into reflectivity by, whenever applicable, taking the mean of the counting rates at angles smaller than the critical angle of total reflection to normalize. That way Reflectivity is a measure that goes from 0 to 1 (alternatively 0-100%). By using the available wavelength of the instrument, the wave-vector magnitude  $q$  can be calculated from the corresponding *omega* angles. The raw data is saved in text file format that can be edited with a text editing software of your choice (e.g. Excel, Origin, Matlab). The structure of the data files will be explained to by the supervisor.

Determine the critical angle of total reflection and the  $q_c$  of the reflectivity curve measurements and compare them with the data from Table 1 in the appendix.

### What material is the layer coating made off?

Use equation (9) to determine the layer thickness of the monolayer (sample 1) in first approximation and estimate the accuracy of the result. Then use equation (10) to get a more accurate result in the layer thickness determination.

Specify the  $d$ -value (thickness of individual layers) of the multilayer (sample 2) and the  $m$ -value of the supermirror (sample 3).

## 6 Written report

The report should include the aim of the experiment as well as a description of the measurement and alignment procedures in your own words. Please also include the result of these alignments by means of the alignment scans (*detarm*, *omega* and *sample\_y*).

Please answer the questions from this guide. The main part of the protocol contains the presentation and the analysis of the measured data. Create reflectivity curves from your measured data ( $R(q)$  or  $R(\theta)$ ) of the different samples. Use equations (9) and (10) to determine the layer thickness of the monolayer (sample 1) and determine from which material is made of, calculate the layer thickness of the multilayer (sample 2) and define the m-value of the supermirror (sample 3).

## 7 Appendix A: Scattering lengths for selected materials.

Material	b [fm]	N [ $10^{28}m^{-3}$ ]	1-n [ $10^{-6}$ ]	$q_c$ [Å]	Remarks
H	-3.73				
D ( $^2H$ )	6.67				
C	6.64	11.3	19.1	0.019	Graphite
C	6.64	17.6	29.8	0.024	Diamond
O	5.80				
Si	4.15	5.00	5.28	0.010	
Ti	-3.44	5.66	-5.0	-	
Fe	9.45	8.50	20.45	0.020	
Co	3.63	8.97	8.29	0.013	
Ni	10.3	9.14	24.0	0.022	
Cu	7.72	8.45	16.6	0.018	
Ag	5.92	5.85	8.82	0.013	
Au	7.63	5.90	11.5	0.015	
H <sub>2</sub> O	-1.68	3.35	-1.43	-	
D <sub>2</sub> O	19.1	3.34	16.2	0.018	
SiO <sub>2</sub>	15.8	2.51	10.1	0.014	Glass
GaAs	13.9	2.21	7.82	0.012	
Al <sub>2</sub> O <sub>3</sub>	24.3	2.34	14.5	0.017	Sapphire

Table 1: The scattering lengths, refractive indexes for  $\lambda = 4 \text{ \AA}$  and critical wave-vectors of selected materials.



## 8 Appendix B: NICOS control commands

<i>Commands</i>	<i>Description</i>	<i>Example</i>
<i>count(preset)</i>	<i>counts for „preset“ seconds</i>	<i>count(2)</i>
<i>move(Axis, time)</i>	<i>run command for a movement</i>	<i>move(omega,0)</i>
<i>read(Axis)</i>	<i>Read the current position</i>	<i>read(sample_y)</i>
<i>adjust(Axis, position to adjust)</i>	<i>Zero point correction of axis</i>	<i>adjust(omega,0)</i>
<i>cscan(Axis,center,step,N,preset)</i>	<i>centered scan around a position „center“ with step size „step“ and „N“ steps on each side</i>	<i>sscscan(omega,0,0.05,7,2)</i>
<i>sscscan(Axis,start,step,N,preset)</i>	<i>Scan from position „start“ with „N“ steps of step size „step“</i>	<i>sscscan(sample_y,-2,0.1,10,2)</i>

*Table 2: Compilation of the most relevant control commands for TREFF in NICOS.*