Institut für Mikro- und Nanomaterialien

PRAKTIKUM
„Werkstoffe der Elektrotechnik“

Versuch B: Röntgenbeugung (XRD)

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1 Theory

1.1 Crystals and their Structure

During the complete history of mankind crystals fascinated humans, because they were rare and something special in nature. Suspected to have divine, demonic or magical powers, gemstones were for the rich and powerful. Jewelry was made of those stones, that were pure and as it seemed without any defects. The word crystal comes from the Greek word κρύσταλλος (krystallos), which means ice or petrified ice in ancient Greek. They used this word also for quartz, found in mines, from which they thought it was water frozen in high altitudes at so low temperature that it will never melt again. People believed this up to the mediaeval times. However, with the development of modern science the view onto crystals changed. Nowadays crystal means a solid material which atoms or molecules are not randomly distributed but regularly ordered. With x-ray diffraction it is possible to analyze its phase composition, the mean grain size and even the structural composition.

Bravais Lattices

In 1849 Auguste Bravais proved that there are only 14 possible ways in three dimensional space to arrange an elemental or unit cell with the following axioms:

1. The unit cell is the simplest repeating unit in a crystal,
2. Opposite surfaces in an unit cell are parallel,
3. The lateral faces of an unit cell connect equivalent positions

These 14 Bravais lattices are the combination of the 7 basic lattices with the possible symmetry options. There are following lattices, with decreasing symmetry:

<table>
<thead>
<tr>
<th>System</th>
<th>Axial Translation</th>
<th>Axial Angles</th>
<th>Picture</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. cubic</td>
<td>a = b = c</td>
<td>α = β = γ</td>
<td>1.1</td>
</tr>
<tr>
<td>2. tetragonal</td>
<td>a = b ≠ c</td>
<td>α = β = γ</td>
<td>1.2</td>
</tr>
<tr>
<td>3. orthorhombic</td>
<td>a ≠ b ≠ c</td>
<td>α = β = γ</td>
<td>1.3</td>
</tr>
<tr>
<td>4. hexagonal</td>
<td>a = b ≠ c</td>
<td>α = β = 90°, γ = 120°</td>
<td>1.4</td>
</tr>
<tr>
<td>5. trigonal</td>
<td>a = b = c</td>
<td>α = β, γ ≠ 90° &lt; 120°</td>
<td>1.5</td>
</tr>
<tr>
<td>6. monoclinic</td>
<td>a ≠ b ≠ c</td>
<td>α = γ = 90°, β &gt; 90°</td>
<td>1.6</td>
</tr>
<tr>
<td>7. triclinic</td>
<td>a ≠ b ≠ c</td>
<td>α ≠ β ≠ γ ≠ 90°</td>
<td>1.7</td>
</tr>
</tbody>
</table>

Table 1: Basic Lattices
**Figure 1: Cubic Lattices**

(a) simple  
(b) body centered  
(c) face centered

**Figure 2: Tetragonal Lattices**

(a) simple  
(b) body centered

**Figure 3: Orthorombic Lattices**

(a) simple  
(b) base centered  
(c) body centered  
(d) face centered
Figure 5: Hexagonal Lattice

Figure 4: Trigonal Lattice

Figure 6: Monoclinic Lattices

Figure 7: Triclinic Lattice
**Miller Indices**

Miller indices form a notation system in crystallography for planes in crystal (Bravais) lattices. A family of lattice planes is determined by three integers $h, k, l$ that are called the Millers indices. They are written $(hkl)$, negative integers are written with a bar over the number. The integers are usually written in the lowest terms, what means their greatest common divisor should be 1. There is a simple procedure to determine the Millers indices of a plane:

- Extend the plane to make it cut the crystal axis system at points $(a_1, a_2, a_3)$
- Note the reciprocals of the intercepts, i.e.: $(1/a_1, 1/a_2, 1/a_3)$
- Multiply or divide by the highest common factor to obtain the smallest integer numbers
- Replace negative integers with bar over the number
- If the plane is parallel to an axis, we say it cuts at $\infty$ and $1/\infty = 0$.
- If the plane passes through the origin, we translate the unit cell in a suitable direction.

**Lattice Planes**

A crystal lattice plane is defined by points in the crystal lattice and its position in space is given by the Miller indices (hkl). It can be described by the linear combination of the three basis vectors $\vec{a}_1$, $\vec{a}_2$ and $\vec{a}_3$. A plane is defined by its intersection with the crystal axes. The Miller indices describe those planes that contain the points $\frac{1}{h}\vec{a}_1$, $\frac{1}{k}\vec{a}_2$ and $\frac{1}{l}\vec{a}_3$.

The planes intercept the axes at the reciprocal value of the indices, whereas an index of 0 means that the intercept is in infinity (the plane is parallel to the basis vector). The reciprocal lattice vector $\vec{G} = h \cdot \vec{g}_1 + k \cdot \vec{g}_2 + l \cdot \vec{g}_3$ is perpendicular on the plane that is defined by the (hkl). The vectors $\vec{g}_1, \vec{g}_2$ and $\vec{g}_3$ are the basis vectors of the reciprocal lattice.

All lattice planes with the same distance $d_{hkl}$ can be calculated by

$$d_{hkl} = \frac{2 \cdot \pi}{h \cdot \vec{g}_1 + k \cdot \vec{g}_2 + l \cdot \vec{g}_3}$$

In Crystal systems with orthogonal axes e.g. rhombohedral, tetragonal and cubic, the following formula is valid for the spacing:

$$d_{hkl} = \frac{1}{\sqrt{h^2 + k^2 + l^2}}$$ (1)

In cubic lattices where $a=b=c$ is essential, there is

$$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$ (2)
1.2 Producing X-Rays

On November 8th 1885 x-rays were detected for the first time by Wilhelm Conrad Röntgen. He produced this high energetic radiation by accelerating electrons in an electric field and letting hit them a target, as shown in figure 10.

By this method, two kinds of radiation are emitted. The electrons hit the atoms of the target and eject electrons of the innermost orbitals. When electrons fall back to this lower energy levels, by filling the gaps produced by the previous process, electromagnetic radiation of specific wavelengths is emitted. See figure 9 for some of the possible electron transitions.

This is the main source for x-rays. The second source is the so called “Bremsstrahlung” (deceleration in German). When accelerated electrons are slowed down abruptly, they also emit a diffuse radiation in the range of the x-rays. Contrary to the first source they have a broad wavelength spectrum.
1.3 X-Ray Diffraction

In 1912 Max von Laue, Walter Friedrich and Paul Knipping found that x-rays are diffracted at crystalline materials. With their experiments they confirmed, that crystals have a regular structure and that x-rays have wave character. In the same year Sir William Lawrence Bragg explored the relationship between the structural parameters of crystal materials and the results of x-ray diffraction patterns. The relation

\[ n \cdot \lambda = 2 \cdot d_{hkl} \cdot \sin(\theta) \]  

(connects the wavelength \( \lambda \) of the radiation with the distance between the crystallographic layers \( d_{hkl} \) and the incident angle with the plane \( \theta \), where \( n \) is the order of diffraction. It is known as Bragg-equation.

For x-ray diffractometry the radiation has to fulfill some premises, it must be Monochromatic, Parallel and Coherent.

Monochromaticity is important, because depending on the wavelength of the radiation the diffraction angle varies, as shown in equation 3. The beam has to be parallel and coherent to allow interference.

When we expose a crystal to x-rays they are absorbed at the atoms of the lattice, so that the electrons are accelerated and each atom emits a spherical wavelet itself, as shown in figure 10.

\[ \text{Figure 10: X-Rays lead to Emission of Spherical Waves} \]

According to Huygens, these spherical wavelets are superposed to create a “reflected” wavefront which is emitted having the same wavelength \( \lambda \) and fulfilling the condition “angle of incidence = angle of reflection”.

Constructive interference occurs when the reflected rays at the individual lattice planes have path differences (\( \Delta \)) that are integral multiples of the wavelength \( \lambda \) (\( \Delta = n\lambda \)). Therefore from figure 11,

\[ \text{Figure 11: Reflection of Rays by two Lattice Planes} \]

where the path difference is equal to \( 2 \cdot d \cdot \sin(\theta) \) one can easily derive Bragg’s law.
1.4 Selection rules

The Bragg's law is a necessary but insufficient condition for diffraction. It only defines the diffraction condition for primitive unit cells, where atoms are only at unit cell corners. For all other crystal structures, the unit cells have atoms at additional lattice sites. These are extra scattering centers that can cause out of phase scattering at certain Bragg angles. Therefore we get some selection rules, which define which diffraction peaks we can see for different lattice structures:

<table>
<thead>
<tr>
<th>Bravais lattice</th>
<th>Example compounds</th>
<th>Allowed reflections</th>
<th>Forbidden reflections</th>
</tr>
</thead>
<tbody>
<tr>
<td>Simple cubic</td>
<td>Po</td>
<td>Any ( h, k, l )</td>
<td>None</td>
</tr>
<tr>
<td>Body-centered cubic</td>
<td>Fe, W, Ta, Cr</td>
<td>( h + k + l = ) even</td>
<td>( h + k + l = odd )</td>
</tr>
<tr>
<td>Face-centered cubic</td>
<td>Cu, Al, Ni, NaCl, LiH, PbS</td>
<td>( h, k, l ) all odd or all even</td>
<td>( h, k, l ) mixed odd and even</td>
</tr>
<tr>
<td>Diamond F.C.C.</td>
<td>Si, Ge</td>
<td>all odd or all even with ( h+k+l = 4n )</td>
<td>( h, k, l ) mixed odd and even, or all even with ( h+k+l \neq 4n )</td>
</tr>
<tr>
<td>Triangular lattice</td>
<td>Ti, Zr, Cd, Be</td>
<td>( l ) even, ( h + 2k \neq 3n )</td>
<td>( h + 2k = 3n ) for odd ( l )</td>
</tr>
</tbody>
</table>

1.5 The Debye-Scherrer Method

The method described in the previous chapters is used for analyzing single crystal materials. In principle this method is the best choice for structure analysis and a generally accepted method in chemistry. Unfortunately it is very difficult to produce a monocrystal. In 1916 Peter Debye and Paul Scherrer developed a method to characterize powder and polycrystalline samples. Powders have some special advantage. The crystals are randomly orientated, so that there are in well mixed samples enough crystallites of the same orientation in Bragg condition.

From a diffractogram several conclusions can be made:

1. The number of lines is characteristic for the crystal system, so they can give hints to the crystal system of the examined sample,
2. The relative position of the peaks is characteristic for each lattice. The d-spacing can be easily calculated and with some further calculations the lattice constants. With these information an identification of the phase can be made. Possible stress in the sample can also be indicated by the position, since stress cause a peakshift,
3. The peak width is a measure for the grain size of the sample (Scherrer equation),
4. The intensity of the lines can be an indication for surface texture
1.6 Edge Absorption

When x-rays pass through matter, they are attenuated by absorption and scattering of the x-ray quanta; the absorption effect often predominates. This is essentially due to ionization of atoms, which release an electron from an inner shell, e.g. the K-shell. This can only occur when the quantum energy \( E = \frac{h \cdot c}{\lambda} \) is greater than the binding Energy \( E_K \) of the shell. Where \( h \) is the Planck's constant, \( c \) is the velocity of light and \( \lambda \) is the wavelength of the X-Ray.

The absorption of the material thus increases abruptly as a function of the X-Ray Photon Energy at

\[
E_K = \frac{h \cdot c}{\lambda_K}
\]

This abrupt change is known as the absorption edge, here the K-absorption edge. See also figure 12, where the absorption of Carbon over the X-Ray Energy is plotted.

![Figure 12: Mass Absorption Coefficient over Photon Energy](image)

1.7 Duane-Hunt Relation

The bremsstrahlung continuum in the emission spectrum of an x-ray tube is characterized by the lower limit wavelength \( \lambda_{\text{min}} \), which becomes smaller as the tube high voltage increases. In 1915, the American physicists William Duane and Franklin L. Hunt discovered an inverse proportionality between the limit wavelength and the tube high voltage:

\[
\lambda_{\text{min}} \sim \frac{1}{U}
\]

This Duane-Hunt relationship can be explained by examining some basic considerations:

An X-Ray Quantum attains maximum energy, when it gains the total kinetic energy, when accelerated by the acceleration voltage \( U \):

\[
E = q \cdot U
\]

(with elementary charge \( q = 1.6022 \cdot 10^{-19} \text{ As} \)). With

\[
E = \frac{h \cdot c}{\lambda}
\]

we find:
\[ \lambda_{\text{min}} = \frac{h \cdot c}{N} \cdot \frac{1}{U} \]

With this we get a linear proportionality between \( \lambda_{\text{min}} \) and \( \frac{1}{U} \).

Figure 13: Emission Spectrum of an X-Ray Tube
1 X-ray-reflectometry (XRR)

1.1 Motivation
Most technological applications of thin films require films of definite thickness. This is because most properties of thin films are thickness dependent. Hence, determination of film thickness with high precision is very crucial for these technologies. XRR is a non-destructive and non-contact technique for thickness determination between 2-200 nm with a precision of about 1-3 Å. In addition to thickness determination, this technique is also employed for the determination of density and roughness of films and also multilayers with a high precision.

1.2 The Principle
XRR method involves monitoring the intensity of the x-ray beam reflected by a sample at grazing angles. A monochromatic x-ray beam of wavelength \( \lambda \) irradiates a sample at a grazing angle \( \omega \) and the reflected intensity at an angle \( 2\theta \) is recorded.

![Diagram of XRR setup](image)

Figure 1: \( \theta/2\theta \)-Scan: The condition of incident angle \( \omega = (2\theta)/2 = \theta \) = outgoing angle is satisfied. The detector D rotates at twice the speed of the sample P. This arrangement is sensitive only to the planes parallel to the surface of the sample. The beam makes an incident angle \( \omega \) with the surface of the sample P. The reflected intensity at angle of \( 2\theta \) is measured. Both the rotation of the sample \( \omega \) and the detector \( (2\theta) \) are about the same axis MP (perpendicular to the drawing). The sample is adjusted so that the rotation axis lies on the sample surface. The Detector circle is fixed through the (programmable) detector slit \( (PRS, \text{programmable receiving slit}) \). The anode focus, \( F \) of the tube lies on the detector circle.
by a detector, see figure 1. This figure illustrates specular reflection where the condition $\omega = 2\theta/2$ is satisfied. The mode of operation is therefore $\theta/2\theta$ mode which make sure the incident angle is always half of the angle of diffraction. The reflection at the surface and interfaces is due to the different electron densities in the different layers (films), which corresponds to different reflective indexes in the classical optics. For incident angles $\theta$ below a critical angle $\theta_c$, total external reflection occurs. The critical angle for most materials is less than $0.3^\circ$. The density of the material is determined from the critical angle. Above $\theta_c$ the reflection from the different interfaces interfere and give rise to interference fringes. The period of the interference fringes and the fall in the intensity are related to the thickness and the roughness of the layer (layers in case of multilayers). The reflection can be analyzed using the classical theory (Fresnel equation). The typical range for these measurements are between $0^\circ$ and $5^\circ$ in $\theta$.

1.2.1 Film density

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

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

(1)

where $\delta$ and $\beta$ represent the dispersion and absorption, respectively. For frequencies far greater than the resonance frequencies, $\vartheta_0$, of the atom $\delta$ can be given by the expression

$$\delta = \frac{e^2 n_e}{2\epsilon_0 m(2\pi e)^2} \lambda^2 = \frac{r_0 \lambda^2}{2\pi} \cdot n_e$$

(2)

where $r_0$ is the Bohr atomic radius and $n_e$ is the electron density. The electron density is given by $n_e = Z \cdot n_{\text{Atom}}$ where $Z$ is the number of electrons per atom. For a more precise expression of $\delta$, $Z$ is usually replaced with the a complex atom form factor $f = f_0 + f' + if'' = Z + f' + if''$. The term $f' + if''$ is due to dispersion and absorption and describes the x-ray absorption edge. It follows that

$$\delta = \frac{r_0 \lambda^2}{2\pi} \cdot (Z + f') \cdot n_{\text{Atom}}$$

(3)

and

$$\beta = \frac{r_0 \lambda^2}{2\pi} \cdot f'' \cdot n_{\text{Atom}}.$$ 

(4)

The magnitudes of $\delta$ and $\beta$ are of the order of $10^{-5}$ and $10^{-6}$, respectively. Since the atomic concentration is given as

$$n_{\text{Atom}} = \frac{N_A}{A} \cdot \rho$$

it is apparent that, the density of the material can be determined from the values of $\delta$ and $\beta$. Here $N_A$ is the Avogadros number and $A$ the atomic weight. The
Figure 2: XRR-measurements and simulations of an Tungsten oxide film on glass. The films were deposited using different deposition parameters. A shift in the total reflection edge is apparent which implies a change in density. The sample with the lowest density (lowest reflection edge) exhibits interference fringes in a shorter angular range than the sample with higher density. This implies that the roughness of the sample with lower density is higher than the roughness of the sample with higher density. The thickness of the samples are obtained from the period of the oscillations.

density of a compound material of known stoichiometry can also be determined from $\delta$ and $\beta$ with slight modifications in the formula.

We illustrate the determination of the different quantities obtained from XRR measurement with an example of a single film on a substrate. For qualitative discussion, it is adequate to consider an absorption free film i.e $\beta = 0$ but it should be noted that $\beta$ cannot be ignored in the simulation of XRR measurements. 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

$$1 - \delta = \cos \theta_c$$

$$\approx 1 - \frac{\theta_c^2}{2}$$

$$\Leftrightarrow \theta_c \approx \sqrt{2\delta}$$

$$= \sqrt{\frac{r_0 \lambda^2}{\pi} N_A \frac{(Z + f')}{A} \cdot \rho}$$

A typical measurement is shown in fig. 2 where the total reflection angle is determined by the abrupt fall in the reflected intensity.
1.2.2 Film thickness

For incident angles greater than \( \theta_c \), \((\theta > \theta_c)\) 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 does not depend on the frequency like in the case of optical spectroscopy but are angle dependent. Due to the low amplitude reflection coefficient \((\rho_{v,h} \sim 1/\sin^2 \theta \Rightarrow \rho_{v,h} = |r_{v,h}| \sim 1/\sin^4 \theta \approx 1/\theta^4)\) of interface between adjacent layers, contributions of multiply reflected beams can be neglected. The m-th interference maximum for a path difference \(\Delta = m \lambda\), is located at

\[
m\lambda = \Delta = 2d\tilde{N}_{X,1}(\theta_m) \approx 2d\sqrt{\theta_m^2 - 2\delta}\ \text{mit } m \in \mathbb{N}.
\]

\[
\Leftrightarrow \theta_m^2 \approx m^2 \frac{\lambda^2}{4d^2} + 2\delta \Rightarrow m^2 \frac{\lambda^2}{4d^2} + 2\delta = \frac{\theta_m^2}{\theta_C^2}.
\]

If the substrate is optically denser than the film, a phase difference of \(\pi\) occurs at the reflection film / substrate interface and \(m\) is substituted with \(m + \frac{1}{2}\). Employing equation (7) and the difference between two neighboring maxima and minima, the thickness can be determined and is given by

\[
d \approx \frac{\lambda}{2} \frac{1}{\sqrt{\theta_{m+1}^2 - \theta_C^2} - \sqrt{\theta_m^2 - \theta_C^2}}
\]

\[
\approx \frac{\lambda}{2} \frac{1}{\theta_{m+1} - \theta_m}, \quad \text{für } \theta_m \gg \theta_C.
\]

The thickness is often determined with a precision better than 1 Å for measurements exhibiting interference fringes in a bigger angular range. The thickness influence is illustrated in fig. 2.

1.2.3 Surface Roughness

Another important quantity determined from XRR measurements is the surface- and interface-roughness. Roughness gives rise to diffuse scattering, resulting to a less intensity in the specularly reflected beam. General scattering formalism have been developed that calculate the scattered fields for both specular and non-specular scattering. In one of the formalism, Névot and Croce considered roughness by assuming non-homogeneous thickness. They assumed that the thickness has a gaussian distribution with a mean \(d\) and a standard deviation \(\sigma\). With this
assumption, they corrected the Fresnel coefficients of reflection $\rho_{v,h}$ as

$$\rho_{v,h} \cdot \exp \left( -\sigma^2 k^2 \right)$$

The influence of roughness is clearly shown in the fig. 2

1.3 Example

The switching kinetics of Tungsten oxide (see optical active films) due to hydrogen presence mainly depend on the porosity of the films. The switching processes for highly porous films are faster than for films with low porosity. By varying the deposition parameters, films of different porosity are sputtered using a locally assembled sputtering unit. XRR technique is used to determine the development of density and roughness of the films and these results are compared with the results of the switching kinetics. An XRR measurement is shown in fig. 2.
2 Questions to Prepare

1) Briefly describe the events that lead to an x-ray diffraction pattern.

2) Briefly describe why we need a filter in an x-ray tube.

3) What is the easiest way to filter x-rays (see 1.6)?

4) Which material would be suitable to filter away the Kβ-line and let the Kα-line pass, for an x-ray tube with a Mo anode?

5) Which material would be suitable to filter away the Kβ-line and let the Kα-line pass, for an x-ray tube with a Cu anode?

6) Cu has face-centered cubic structure. The lattice constant is 3.6149 Å. Calculate the position of the first five diffraction peaks, when the x-ray wavelength is 1.540 Å (Cu Kα anode).

7) You obtain a diffractogram of a powder material. After annealing it for a short period (without changing the phase of the material) you obtain a second diffractogram. What would you expect for the peaks’ shape? Why?


3 Experiment

As described in the previous chapters, diffraction of x-rays at crystals can be used to characterize materials. One of the biggest application fields of this method is the phase analysis; in geology it is the basic method to analyze rock cuttings. In this lab course the following experiments will be performed:

1. Edge Absorption: Filtering X-rays,
2. Duane-Hunt Relation and determination of Planck’s constant
3. Bragg Reflection: Diffraction of X-Rays at a monocrystal,
4. Debye-Scherrer Scan: determination of the lattice-plane spacing of a polycrystalline sample

Some hints for the report:

• Give a short introduction to the given tasks. Use your own words!
• Answer the questions from the question part in understandable sentences.
• Show pictures wherever it is possible and helpful. A picture is worth a thousand words!
• Add the diffractograms after the text and refer to them in the text.
3.1 Investigation of the Energy Spectrum of the X-Ray Tube

This experiment records the energy/wavelength spectrum of an x-ray tube with a copper anode. A goniometer with a NaCl crystal and a Geiger-Müller counter tube in the Bragg configuration together comprise the spectrometer. The crystal and counter tube are pivoted with respect to the incident x-ray beam in 2θ coupling (see figure 14).

With Bragg’s law of reflection (equation 3), the wavelength dependency of the X-Rays can be calculated from the scattering angle θ in the first order of diffraction from knowing the (200) lattice plane spacing of NaCl $d_{200}=282.01$ pm:

$$\lambda = 2 \cdot d_{200} \cdot \sin(\theta)$$

Knowing the relationship between energy and wavelength for electromagnetic radiation, the energy distribution of the X-Radiation can be calculated:

$$E = \frac{h \cdot c}{2 \cdot d_{200} \cdot \sin(\theta)}$$

Edge Absorption: Filtering X-Rays

This experiment measures the spectrum of an x-ray tube with copper (Cu) anode, both unfiltered and filtered, using a nickel (Ni) foil.

A goniometer with NaCl crystal (2) and a Geiger-Müller counter tube (3) in the Bragg configuration are used to record the intensities as a function of the wavelength. The crystal and counter tube are pivoted with respect to the incident x-ray beam in 2θ coupling, i.e. the counter tube is turned at an angle twice as large as the crystal (see figure 14). In accordance with Bragg's law of reflection, the scattering angle θ in the first order of diffraction corresponds to the wavelength $\lambda = 2 \cdot d_{hk\ell} \cdot \sin(\theta)$.

**Figure 14: Principle of Diffraction of X-Rays at a Monocrystal. With Filter (4), 2θ between Counter Tube (3) and Incident Beam from Collimator (1), θ between incident beam and Monocrystal (2)**

**Measurement:**

Set the tube voltage $U=30.0$ kV, the emission current $I=1.00$ mA and the step width $\Delta\theta=0.1$ °. Press the COUPLED key to activate 20 coupling of target and sensor and set the lower limit of the target angle to $3^\circ$ and the upper limit to $20^\circ$. Set the measuring time per angular step to $\Delta t=1$ s.

Start measurement and data transfer to the PC by pressing the SCAN key. When the scan is finished, mount the Ni foil supplied with your x-ray apparatus on the collimator (or the detector → where is the difference?) and start a new measurement by pressing the SCAN key.

When you have finished measuring, save the measurement series under an appropriate name by pressing the F2 key. To display the measurement data as a function of the wavelength $\lambda$, open the "Settings" dialog with F5, and in the tab "Crystal", choose "NaCl".
3.2 Duane-Hunt Relation and Determination of Planck's constant

Remove the Ni-Filter and set the emission current $I=1.00 \ mA$, the measuring time per angular step $\Delta t=5 \ s$ and the angular step width $\Delta \theta=0.1^\circ$. Press the COUPLED key to activate 20 coupling of target and sensor and set the lower limit of the target angle to $2^\circ$ and the upper limit to $7^\circ$. Keep the crystal calibration. Measure for acceleration voltages $U=20kV, 23kV, 25kV, 30kV, 35kV$.

To show the wavelength-dependency, open the “Settings” dialog, with F5 and enter the lattice-plane spacing for NaCl. When you have finished measuring, save the measurement series under an appropriate name by pressing the F2 key.

**Evaluation:**

Determine the limit wavelength $\lambda_{\text{min}}$ of every wavelength-spectrum. Then draw a plot of $\lambda_{\text{min}}$ over $\frac{1}{U}$. Determine the slope and compare it with the literature values.
3.3 Bragg Reflection: Diffraction of X-Rays at a Monocrystal

In this experiment, we verify Bragg’s law of reflection by investigating the diffraction of x-rays at a NaCl monocrystal in which the lattice planes are parallel to the cubic surfaces of the unit cells of the crystal. The lattice spacing $d_{200}$ of the cubic face-centered NaCl crystal is half the lattice constant $a_0$. We can thus say $2 \cdot d_{200} = a_0 = 564.02$ pm.

The experiment is performed in the same configuration as shown in figure 14 in chapter 3.1.

Set the x-ray high voltage $U=35.0$ kV, emission current $I=1.00$ mA, measuring time per angular step $\Delta t = 1$ s and angular step width $\Delta \theta = 0.1^\circ$. Undo the crystal calibration (open the "Settings" dialog with F5, and in the tab "Crystal", unselect "NaCl"). Mount the Ni filter.

Press the COUPLED key on the device to enable 2θ coupling of the target and sensor; set the lower limit value of the target angle to $10^\circ$ and the upper limit to $60^\circ$. Press the SCAN key to start the measurement and data transmission to the PC. When the measurement is finished, save the measurement series to a file under a suitable name using the F2.

**Evaluation:**

Evaluate the measured Diffractogram by calculating the lattice plane from the angle of the peak position. Compare with the literature.

3.4 Debye-Scherrer Scan: Determination of the Lattice-plane spacing of a Polycrystalline Material

In this experiment, Debye-Scherrer scans of a polycrystalline material (copper coin) are taken. The Bragg angles for different h, k, l indices are recorded and compared to the calculated values.

There are two different ways to record the spectra with the x-ray apparatus:

- keeping the sample at a fixed angle and let only the sensor rotate
- moving sample and sensor in a coupled motion, similar to the ordinary single crystal scans

Rotating the sample should not make any difference at all, but for geometrical reasons of the non-circular sample the second way is preferred. Also, rotating the sensor alone would give the angle 20, in contrast to coupled rotation, where the X-ray apparatus displays the angle.

Set the tube high voltage to $U=35.0$ kV and the emission current $I=1.0$ mA. Set the time per angular step $\Delta t = 2$ s and the angular step width $\Delta \theta = 0.1^\circ$. Select “coupled”, set appropriate start and stop angles $\theta$ for the scan. Measuring from $\theta = 20^\circ$ to $50^\circ$ is sufficient. Switch on the high voltage and press the automatic scan button for recording the spectra.

**Evaluation:**

- Evaluate the peaks of the diffraction spectrum. Which are the lattice planes that are producing the various peaks?
- Calculate the lattice constant of your sample and compare it with literature
- Comment