Tutorial instructions:
X-ray diffraction on crystals (RBE)
"Fortgeschrittenenpraktikum"

Supervisors: A. Hinderhofer, G. Duva, M. Hodas

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1 Duration

During this tutorial, films with three different samples (copper, tungsten, NaCl) will be exposed and developed. The experimental work will consist of radiation safety instructions (approx. 15 min), alignment of the samples (15 min each), insertion of the films (10 min each), exposure of the X-ray films (90 min each) and the development of the films (20 min each).

2 Goal

Determine the lattice type and lattice constants of each material with the help of their respective diffraction patterns. The lattices are either body-centered cubic (bcc) or face-centered cubic (fcc). The lattice constants can be obtained with high precision (standard deviation of about 0.1%).

In this tutorial, a setup was chosen where X-rays are diffracted by a powder sample and then detected with a photographic film. This technique is called "Debye-Scherrer diffraction".

3   Fundamentals

3.1   Radiation safety

Name	Formula	Unit	Description
Activity		$1\,\mathrm{Becquerel} = 1\,\mathrm{Bq}$	Radioactivity events per second
Energy dose	$D = \frac{\mathrm{d}W}{\mathrm{d}m}$	$[D] = 1\,\frac{\mathrm{J}}{\mathrm{kg}} = 1\,\mathrm{Gy} = 1\,\mathrm{Gray}$	Deposited amount of energy per kg
Dose rate	$\dot{D} = \frac{\mathrm{d}D}{\mathrm{d}t}$	$[\dot{D}] = 1\,\frac{\mathrm{J}}{\mathrm{kg \cdot s}} = 1\,\frac{\mathrm{Gy}}{\mathrm{s}}$	Absorbed dose per second
Equivalent dose	$H = Q \cdot D$	$[H] = 1\,\mathrm{Sv} = 1\,\mathrm{Sievert}$	The equivalent dose is dependent on the quality factor Q. For X-rays $Q \approx 1$.

Table 1: Quantities and units in radiation safety

Every participant will receive a dosimeter. The displayed value (mSv) will be recorded before and after the experiment. Areas with a maximum yearly dose below $1\,\mathrm{mSv}$ are freely accessible. This means for an experiment with a duration of 5 hours, the maximum allowed equivalent dose is $2.5\,\mu\mathrm{Sv}$ per year (assuming 40 work hours per week).

Before turning on the X-ray tube, a ratemeter with an acoustic signal is placed next to the tube. The ratemeter remains active during the whole experiment. If there is a change in the number of signals per unit time, the setup is leaking radiation and the X-ray tube must be turned off immediately by pressing the red button in the middle. In that case, please do not perform any further experiments and inform your supervisor immediately.

3.2   Origin of X-ray radiation

Like visible light and γ-radiation, X-rays can be viewed as electromagnetic waves or photons (particle-wave dualism). The electromagnetic spectrum reaches from radiowaves (long, medium, short and ultrashort waves) to microwaves, infrared light, visible light (between ca. 750 and 400 nm), ultraviolet light, X-rays and γ-radiation; with decreasing wavelength λ and (due to $c = \lambda f$ and $E = hf$) increasing photon energy E (see Figure 1).

However, the X-ray spectrum shows some overlap with the γ-radiation spectrum. The wavelengths of X-rays range from $8 \cdot 10^{-8}\,\mathrm{m} = 80\,\mathrm{nm}$ to $10^{-14}\,\mathrm{m}$, which corresponds to frequencies from $4 \cdot 10^{15}\,\mathrm{Hz}$ to $3 \cdot 10^{22}\,\mathrm{Hz}$ and photon energies from $15\,\mathrm{eV}$ to $150\,\mathrm{MeV}$. In comparison, γ-rays have wavelengths smaller than $10^{-11}\,\mathrm{m}$, which corresponds to frequencies above $3 \cdot 10^{19}\,\mathrm{Hz}$ and photon energies above $100\,\mathrm{keV}$.

3.2.1   (Continuous) X-ray "Bremsspektrum" and (discrete) characteristic X-ray spectrum

When electrons with a high energy (e.g. β-rays or electrons in an X-ray tube) hit matter, they are accelerated by the electric field of the atomic nucleus. According to classical electrodynamics, every accelerated electrical charge emits energy in form of electromagnetic
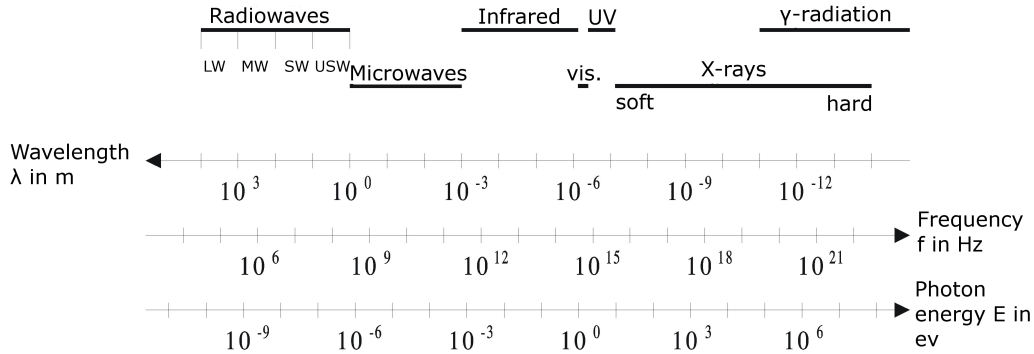
Figure 1: Electromagnetic spectrum. Vis. refers to the spectrum of visible light while UV refers to ultraviolet light. Please note the logarithmic scale.

radiation. This means that the electron must become slower due to a loss of energy. Depending on the amount of energy lost during this process, the emitted X-ray photon has a different energy. Since the electron can lose any amount of energy, the resulting "Bremsspektrum" must be a continuous spectrum of X-rays with maximum wavelength of λ_{max}, which corresponds to a complete loss of kinetic energy. If the kinetic energy of the electrons is only created by applying a voltage U, the maximum wavelength is given by

$$\lambda_{\mathrm{max}} = \frac{c}{f_{\mathrm{max}}} = \frac{hc}{E_{\mathrm{kin}}} = \frac{hc}{eU} \ . \tag{1}$$

When it hits matter, a fast electron can also ionize an atom by ejecting one of the electrons of the inner shells. Since the inner shells are energetically more favorable than the outer shells, the free spot within the inner shell will then be occupied by one of the outer-shell electrons. The excess energy gained through the energy difference between the two states will also be emitted in the form of electromagnetic radiation. Since the energy difference between the shells is quite high, the resulting radiation will be in the range of X-rays, but restricted to the discrete energy differences between the shells of the atom. For that reason, the resulting discrete X-ray spectrum is also called "characteristic spectrum", since it is characteristic for every chemical element.

Every time fast electrons hit matter, both of these processes occur: the emittance of Bremsstrahlung radiation and characteristic radiation. This means the measured X-ray spectrum is always a superposition of a continuous spectrum and characteristic lines.

Wavelength [nm]	Description
0.154178	Average of the K_α double line
0.1540562	Cu $K_{\alpha 1}$
0.1544390	Cu $K_{\alpha 2}$
$\frac{CuK_{\alpha 1}}{CuK_{\alpha 2}} = \frac{2}{1}$	Intensity ratio between the two components

Table 2: Wavelengths and intensity ratios of Cu K_α radiation

3.3   Interaction between matter and electromagnetic radiation

When a photon passes through matter, the following processes can contribute to a reduction of the intensity of the primary beam.

1. Classical scattering (σ_k)

2. Photoelectric effect (τ)

3. Compton effect (σ_C)

4. Pair production (κ)

Classical scattering: When a photon hits a shell electron and the recoil is absorbed by the whole atom, the electron remains in the shell and creates collective electromagnetic oscillations together with all other electrons. In turn, these oscillating electrons emit a photon with the same energy as the absorbed photon, but generally in a different direction than the primary beam.
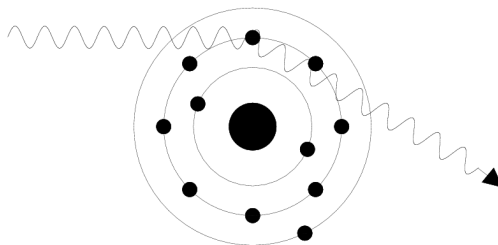

Figure 2: Classical scattering

The mass attenuation coefficient, σ_k/ρ, caused by classical scattering is approximately a linear function of the photon energy E_γ (in the range $10\,\text{keV} \leq E_\gamma \leq 20\,\text{keV}$), the atomic number Z and the mass number A of the absorber material:

$$\sigma_k/\rho \sim \frac{Z^{2.5}}{A}\,1/E_\gamma^2 \tag{2}$$

Photoelectric effect: The term photoelectric effect describes the case where the photon transfers all of its energy to the atom, which then emits an electron. This type of electron is commonly called photoelectron. The energy of the emitted electron is equal to the difference between its former binding energy in the atom and the photon energy. For low photon energies, the attenuation of the X-ray beam is mainly caused by the photoelectric effect.

The dependence of the mass attenuation coefficient on E_γ, Z and A caused by the photoelectric effect, τ/ρ, can approximately be described by

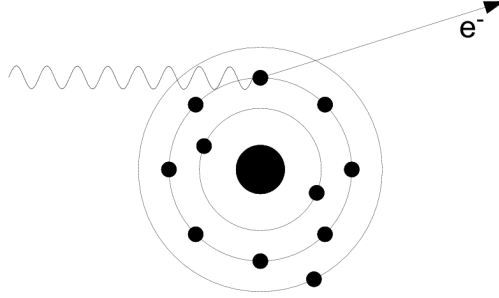$$\tau/\rho \sim \frac{Z^4}{A}\,1/E_\gamma^3 \tag{3}$$

Figure 3: Photoelectric effect

Compton effect: The elastic scattering of a photon with a quasi-free electron is called Compton scattering. During this process—similar to the elastic collision of two spheres with different masses—the photon transfers part of its energy to the electron, which is then ejected from the atom. This leads to an ionization of the atom. After this interaction, the energy E_x of the scattered photon is reduced by the amount of the transfered energy E_k:

$$E_x = E_\gamma - E_k, \tag{4}$$
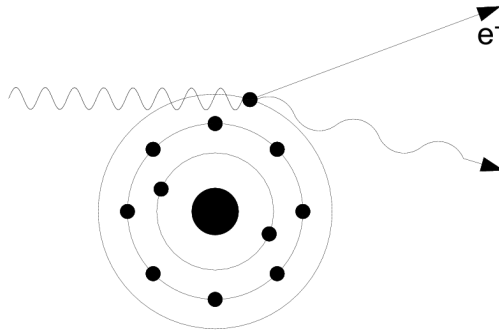
where E_γ is the energy of the incoming photon.

Figure 4: Compton effect

Between 0.2 and 10 MeV, the dependence of the mass attenuation coefficient on E_γ, Z and A caused by Compton scattering, σ_C/ρ, can approximately be described by

$$\sigma_C/\rho \sim \frac{Z}{A}\, 1/E_\gamma^{1/2} \tag{5}$$

Pair production: Above a quantum energy of about 1.1 MeV, another process, called pair production, is possible. During this process, the photon within the Coulomb field of the atomic nucleus is converted into an electron-positron pair. Positrons are antiparticles to electrons and have the exact same properties except that they have the opposite

(positive) charge. The sum of the resting energies[1] of the two particles is $2 \cdot 511 \, \text{keV}$ $= 1.022 \, \text{MeV}$. This means pair production is only possible for photon energies higher than that amount. For high photon energies (above ca. $60 \, \text{MeV}$ for elements with a small Z), the attenuation of the X-ray is mainly caused by pair production.
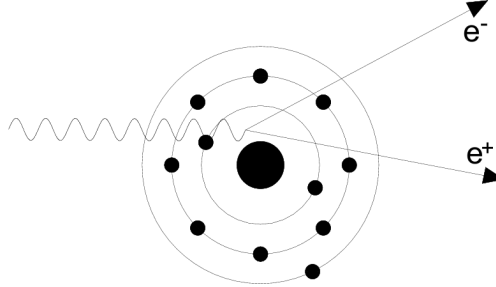

Figure 5: Pair production

The dependence of the mass attenuation coefficient on E_γ, Z and A ($E_\gamma \geq 1.022 \, \text{MeV}$) caused by pair production, κ/ρ, can approximately be described by

$$\kappa/\rho \sim \frac{Z^2}{A} \log(E_\gamma) \tag{6}$$

3.3.1 The law of attenuation for electromagnetic radiation, e.g. light, X-rays and γ-rays

The law of attenuation[2] of electromagnetic radiation states that monoenergetic radiation that passes through layers of matter with equal and infinitesimal thickness is reduced by the same amount μ:

$$\frac{\mathrm{d}I}{I} = -\mu \mathrm{d}x \tag{7}$$

$\mathrm{d}x$ is the infinitesimal layer thickness and μ is called **linear attenuation coefficient**. For finite layer thicknesses x, the transmitted intensity $I(x)$ of electromagnetic radiation with a primary intensity of I_0 can be obtained by integrating Equation 7:

$$I(x) = I_0 e^{-\mu x} \tag{8}$$

Alternatively, this can be expressed with the area mass density $D = \frac{m}{A} = \rho x \, [\text{g/cm}^2]$ instead of the layer thickness and the mass attenuation coefficient instead of the linear attenuation coefficient:

$$I(D) = I_0 e^{-(\mu/\rho)D} \tag{9}$$

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[1]In combination with the relation between the frequency f and energy E of a photon, $E = hf$ (h is the Planck constant), the renowned formula $E = mc^2$ gains even more importance: a single photon with the frequency f can create two (conservation of charge) particles with the mass m and vice versa.

[2]Also called Beer–Lambert law.

The layer thickness at which the intensity of the radiation is reduced to half of its original value is called **half-value thickness**, $d_{1/2}$. It relates to the linear attenuation coefficient μ with

$$d_{1/2} = \frac{\ln 2}{\mu} \tag{10}$$

The individual components of the above mentioned attenuation processes are designated by their respective cross sections: σ_{k} in the case of classical scattering, τ in the case of photoabsorption, σ_{C} in the case of Compton scattering and κ in the case of pair production. The total attenuation is the sum of all cross sections, i.e. the linear attenuation coefficient μ:

$$\mu = \sigma_{\mathrm{k}} + \tau + \sigma_{\mathrm{C}} + \kappa \tag{11}$$

The contribution of each component to the linear attenuation coefficient depends on the energy of the γ-radiation and the atomic number of the matter the radiation is passing through (see subsection 3.3). Figure 6 shows the different components of the mass attenuation coefficient of copper. At small photon energies, photoabsorption is dominant, whereas at energies above $1.2\,\mathrm{MeV}$, attenuation is mainly governed by pair production.
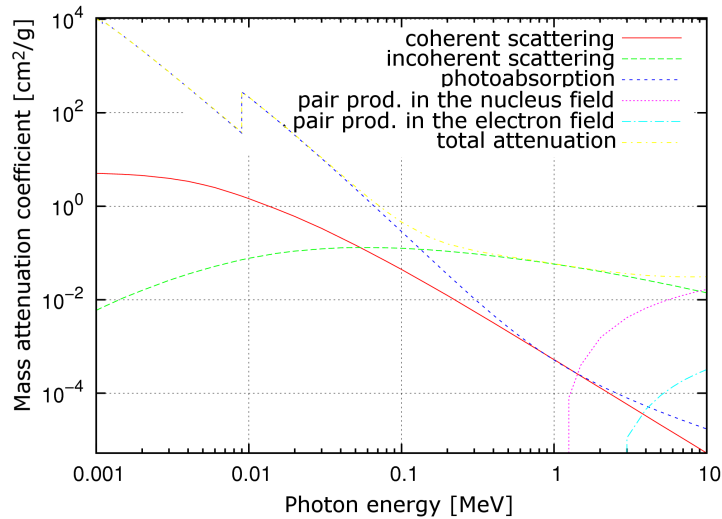

Figure 6: The individual components of the mass attenuation coefficient as a function of the photon energy for Cu ($Z = 29$). Source: NIST Standard Reference Database

3.4   Diffraction on a lattice

According to the Huygen-Fresnel principle, electromagnetic waves that are emitted from a scatterer can be described as spherical waves. The optical path difference between waves from different scatterers is critical for understanding diffraction. The summation of all waves with respect to their phase results in a total wave with an intensity distribution that can later be seen on the developed films at the end of the experiment. The following sections briefly explain the scattering amplitudes for different scattering geometries.
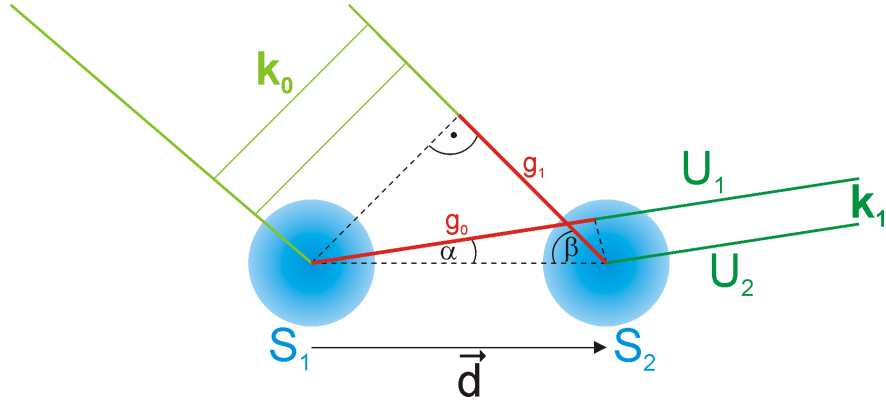
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3.4.1 Diffraction with two scatterers

Figure 7: Diffraction of a plane wave by two scatterers

In the scattering geometry depicted in Figure 7, a plane incoming wave with an angle β is scattered by the two scatterers S_1 und S_2 and outgoing waves with an angle α are emitted. Since we assume elastic scattering, the absolute value of the wave vectors stays constant during the scattering process, i.e. $|\vec{k_0}| = |\vec{k_1}| = 1/\lambda$.[3] The amplitudes of the two scattered waves can be described by

$$U_1 = U_0 e^{i(2\pi \vec{k_1}\vec{r} - \omega t)} \tag{12}$$

$$U_2 = U_0 e^{i(2\pi \vec{k_1}\vec{r} - \omega t + \phi)} \tag{13}$$

where U_2 has a phase difference ϕ to U_1. This means the total amplitude is given by

$$U_{\text{tot}} = U_1 + U_2 = U_1(1 + e^{i\phi}) = U_0 e^{i(2\pi \vec{k_1}\vec{r} - \omega t)}(1 + e^{i\phi}) \tag{14}$$

and its corresponding intensity is

$$I = U_{\text{tot}}U_{\text{tot}}^* = U_0^2(2 + e^{i\phi} + e^{-i\phi}) = U_0^2(2 + 2\cos(\phi)) \tag{15}$$

As a result, the scattering intensity has its maxima at $\phi = 2\pi n$ and its minima at $\phi = \pi(2n + 1)$.

Assignment: Show that the maximum constructive interference (phase difference of ϕ) occurs when $\vec{d} \cdot \vec{q} = n$ ($n \in \mathbb{Z}$). The wave vector $\vec{q}$ is defined by $\vec{q} := \vec{k_1} - \vec{k_0}$ (see Figure 8). The Bragg equation for powder diffraction is a direct result of the definition of the scattering vector and is the connecting piece between the theoretical scattering vector and the experimentally determined scattering angle θ. Figure 8 shows that

$$\sin(\theta) = \frac{|\vec{q}|}{2 \cdot \frac{1}{\lambda}} \tag{16}$$

must be true. The scattering intensity reaches its maximum at $\vec{d} \cdot \vec{q} = n$.

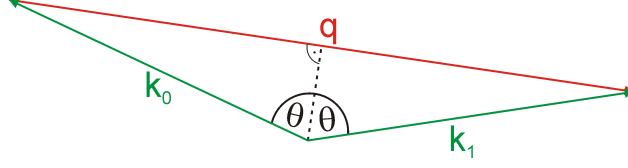[3]Here, we use the crystallographic convention.

Figure 8: Scattering triangle

3.4.2 Diffraction with multiple scatterers

The above considerations can also easily be applied to systems with more than two scatterers. Analogous to the previous case, the wave emitted by the nth scatterer is given by

$$U_n = U_1 e^{i\phi_n} \tag{17}$$

The total amplitude is then the sum of all N scatterers:

$$U_{\text{tot}} = \sum_{n=1}^{N} U_n = \sum_{n=1}^{N} U_1 e^{i\phi_n} = U_1(1 + e^{i\phi_1} + e^{i\phi_2} + ...)$$
$$= U_1(1 + e^{2\pi i \vec{q} \vec{d_1}} + e^{2\pi i \vec{q} \vec{d_2}} + ...) \tag{18}$$

3.4.3 Diffraction with periodically ordered scatterers

If the scatterers are ordered periodically, like in a crystal lattice, the vectors $\vec{d_n}$ can be expressed with help of a base:

$$\vec{d_\nu} = \nu_1 \vec{a_1} + \nu_2 \vec{a_2} + \nu_3 \vec{a_3} \tag{19}$$

with $\vec{a_j}$ being the base vectors and ν_j the coordinates of every scatterer. Additionally, we can define a reciprocal base[4], which describes the position of the scattering vectors $\vec{q}$ in reciprocal space:

$$\vec{q} = h \vec{a_1}^* + k \vec{a_2}^* + l \vec{a_3}^* \tag{20}$$

A summation over the coordinates of every scatterer, while representing $\vec{q}$ and $\vec{d_n}$ by their bases, leads to a total scattering amplitude of

$$U_{\text{tot}} = U_1 \sum_{\nu_1} \sum_{\nu_2} \sum_{\nu_3} e^{2\pi i (\nu_1 h + \nu_2 k + \nu_3 l)} \tag{21}$$

By choosing the reciprocal base correctly, the scalar products in the exponential functions can be simplified significantly.

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[4]The components h, k and l are called Miller indices.

3.4.4   Properties of the reciprocal lattice

The reciprocal base is chosen in such way that it fulfills the relation

$$\vec{a_i} \cdot \vec{a_j^*} = \delta_{ij} \tag{22}$$

The transformation of the real lattice into the reciprocal lattice and vice versa is then given by

$$\vec{a_1^*} = \frac{\vec{a_2} \times \vec{a_3}}{\vec{a_1} \cdot (\vec{a_2} \times \vec{a_3})}, \quad \vec{a_2^*} = \frac{\vec{a_3} \times \vec{a_1}}{\vec{a_1} \cdot (\vec{a_2} \times \vec{a_3})}, \quad \text{and} \quad \vec{a_3^*} = \frac{\vec{a_1} \times \vec{a_2}}{\vec{a_1} \cdot (\vec{a_2} \times \vec{a_3})} \tag{23}$$

This means for a highly symmetrical cubic lattice ($a_1 = a_2 = a_3 := a$)

$$|\vec{a_1^*}| = \frac{a^2}{a^3} = \frac{1}{a}, \quad |\vec{a_2^*}| = \frac{a^2}{a^3} = \frac{1}{a} \quad \text{and} \quad |\vec{a_3^*}| = \frac{a^2}{a^3} = \frac{1}{a} \tag{24}$$

In this case, the absolute value of the scattering vector can be expressed by

$$|\vec{q}| = \sqrt{\frac{h^2}{a^2} + \frac{k^2}{a^2} + \frac{l^2}{a^2}} = \frac{1}{a}\sqrt{h^2 + k^2 + l^2} \tag{25}$$

Since $|\vec{q}|$ is the measured quantity of the experiment, the lattice constant of the real lattice can be determined via the Bragg equation. Combining $|\vec{q}|$ into Equation 16 yields a lattice constant of

$$a = \frac{\lambda}{2\sin(\theta)}\sqrt{(h^2 + k^2 + l^2)} = \frac{\lambda}{2\sin(\theta)}\sqrt{N} \tag{26}$$

For crystal lattices with a lower symmetry, these expressions are much more complex.

3.4.5   Structure factor

Bragg's law lets us make predictions about the angle θ at which diffraction leads to constructive interference, but it does not provide any information about the intensity $I(\theta)$ of the interference maxima. To calculate this intensity, we need the concept of scattering amplitudes.

Within the periodic structure of a crystal lattice, the position of every scatterer can be described by two vectors: the position of the unit cell $\vec{R_n}$ and the position within that unit cell $\vec{r_j}$. The unique position of every scatterer within the crystal is therefore defined by the superposition

$$\vec{R_n} + \vec{r_j} \tag{27}$$

This factorizes the scattering amplitude into a product of two sums:

$$U_{\text{tot}} = U_1 \sum_{r_j} f_j(\vec{q}) e^{2\pi i \vec{q} \cdot \vec{r_j}} \sum_{\vec{R_n}} e^{2\pi i \vec{q} \cdot \vec{R_n}} \tag{28}$$

The first sum is called the structure factor. It contains the phase information of all scatterers within one unit cell, i.e. its crystal structure. The second sum corresponds to the phase information of the crystal lattice, i.e. between different unit cells. This term effectively amplifies the signal of the structure factor. The additional factor $f_j(\vec{q})$ is called the atom form factor and describes the fact that scatterers can have arbitrary shapes. It is obtained by Fourier-transforming the electron density of an atom and is characteristic for every atom species. It is defined by

$$f_j(\vec{q}) = \frac{1}{V_{\text{cell}}} \int_{V_{\text{cell}}} \rho_0(\vec{x}) e^{2\pi i \vec{q} \vec{x}} \mathrm{d}\vec{x} \tag{29}$$

This means when taking Equation 22 into account, the resulting scattering amplitude is given by

$$U_{\text{tot}} = U_1 \sum_\mu f_\mu e^{2\pi i (x_\mu h + y_\mu k + z_\mu l)} \sum_{\nu_1} \sum_{\nu_2} \sum_{\nu_3} e^{2\pi i (\nu_1 h + \nu_2 k + \nu_3 l)} \tag{30}$$

If the unit cell consists only of a single atom, the origin of the position vector $\vec{r_j}$ can be set to the position of the atom, i.e. $x_1 = y_1 = z_1 = 0$. Since this means the index $\mu = 1$, Equation 30 can be simplified to

$$U_{\text{tot}} = U_1 \sum_{\nu_1=0}^{N_1} \sum_{\nu_2=0}^{N_2} \sum_{\nu_3=0}^{N_3} f_1(\vec{q}) e^{2\pi i (\nu_1 h + \nu_2 k + \nu_3 l)}. \tag{31}$$

Analogously, for a base with two atoms $\mu = 1, 2$ which results in

$$U_{\text{tot}} = U_1 \sum_{\mu=1}^{2} \sum_{\nu_1=0}^{N_1} \sum_{\nu_2=0}^{N_2} \sum_{\nu_3=0}^{N_3} f_\mu(\vec{q}) e^{2\pi i (\nu_1 h + \nu_2 k + \nu_3 l)} e^{2\pi i (x_\mu h + y_\mu k + z_\mu l)}. \tag{32}$$

4 Experiment

4.1 Setup

A powder sample in the form of a wire is placed into the camera axis and surrounded by a 35 mm high X-ray film. The collimator and the beam attenuator are placed perpendicular to the camera axis. To enable diffraction for as many crystal grains as possible, the sample is rotated along the camera axis during the measurement.
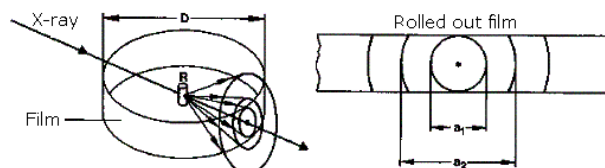

Figure 9: Scattering geometry of a Debye-Scherrer experiment

4.2 Procedure

During this tutorial, three samples will be measured: tungsten, copper and sodium chloride, respectively. Each sample is carefully aligned, measured and its film developed. Afterwards, the films will be analyzed with precision ruler.

4.3 Alignment

First, each wire is placed on an optical "goniometer" and aligned to the axis of the rotatable "goniometer head". This is the most crucial step of the experiment, since the rotating sample should never move out of its rotation axis by more than its own diameter, otherwise the lines will become too broad. The alignment is then tested by exposing the rotating sample to the X-ray beam (without a film) while observing the silhouette of the wire through a leaded glass window. In the dark room, the sample holder will be assembled and loaded with the film. It is recommended to proceed as follows:

1. Insert the film into the film cylinder.

2. Attach the collimator and the primary beam guide and check if the film is positioned correctly. Remove both parts again to avoid hitting them with the sample.

3. Carefully attach the turntable with the aligned goniometer head. Touching any part of the chamber misaligns the sample.

4. Attach the collimator and the primary beam guide again.

5. Close the lid of the chamber.

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4.4   Measurement

The supervisor will turn on the X-ray tube and give instructions on radiation safety. Before activating the X-ray tube, the cooling water supply needs to be turned on. The tube is operated at a voltage of 40 kV and a current of 30 mA.

Material	Exposure time
Tungsten	90 min
Copper	90 min
NaCl	90 min

Table 3: Samples and their exposure times

4.5   Photographic development of the film

The starting point for the development process are the silver nuclei that are created when the silver halide crystals are exposed to light. During this process, a small part of the silver ions is transformed into metallic silver. Silver ions that were not converted will move towards the silver nuclei during the development of the film and become chemically reduced to metallic silver. Since a single silver halide crystal contains up to 1000 silver ions, the development of the film leads to an amplification of the latent image by a factor of a billion.

The developed photographic emulsion still contains light-sensitive silver halide in all areas that were not exposed. To stabilize the image, this silver halide needs to be removed. This is done by submerging the film in a fixing bath and afterwards washing it in a water bath. The fixing bath first creates hardly soluble double salts. The turbidity of the solution disappears after a certain residence time. The solution then forms highly soluble silver thiosulfate complexes, which can be removed by washing the film with water. The most common fixing baths contain either sodium thiosulfate (normal fixing baths) or ammonium thiosulfate (quick fixing baths). Colloquially, the term "development" describes the entire processing of the film, including the fixation, washing and drying steps.

Please note: Do not put anything that came into contact with the fixing bath into the photographic developer or it will become unusable!

Procedure	Duration (min)	
Development	5	Left bath
First wash	1/2	Middle bath
Fixing bath	3	Right bath
Second wash	15	Far right bath
Drying	20	String

Table 4: Development of the exposed X-ray film

5 Analysis

5.1 Diffraction angle

The diffraction angle is determined with a ruler. This is done by measuring the distance between two opposing intensity maxima of the same order. The radius of the Debye-Scherrer chamber is $r = 28.65\,\mathrm{mm}$, which means that a distance of $1\,\mathrm{mm}$ on the film corresponds to a diffraction angle of $1°$.

5.2 Indexing

The Bragg equation allows us to calculate the lattice constants of a crystal lattice if we know the angle and the index triple h, k, l of every diffraction maximum. The process of finding the correct index triple is also called "indexing a diffraction pattern". For unknown compounds, this is the most difficult step of solving the crystal structure of powder samples and does not always lead to unambiguous results. In our case, the diffraction pattern can be clearly indexed by comparing the pattern with predictions from theoretical model calculations. The calculations are performed for body-centered and face-centered cubic crystal lattices. The different lattice types have different translation vectors within their unit cell, which leads to an extinction of diffraction maxima, even if they have a valid index triple h, k, l. These missing maxima can be described by an "extinction rule" which is characteristic for every lattice type.

5.3 Calculation of the lattice constants and their error

If the indexing and the diffraction angles are known, the lattice constants can be calculated:

1. First, calculate the average $\bar{a}$ of all individual lattice constants a_{hkl} and calculate the standard deviation via the error of the angle measurement.

2. One can show that the systematic error of the lattice constants vanishes for $\theta \to 90°$. This means that the lattice constant can be "refined" by plotting a_{hkl} against $\cos^2(\theta)$ and linearly extrapolating to $\cos^2(\theta) = 0$.

5.4 Comparison with the literature

The experimentally determined lattice constants $\bar{a}$ and $a(\theta \to 90°)$ should be compared to values from the literature. It is recommended to use the ICSD database, which can be accessed through http://icsd.fiz-karlsruhe.de. Under the section "Advanced search & retrieve $\to$ Chemistry", different elements can be entered into the search bar. Select the article and click "Show detailed view". When referencing values from the literature, please always provide the journal name and the article ID number. Since the database uses IP authentication, it can only be accessed through the university network (university PC, WiFi or VPN). Please do not use any references from Wikipedia or similar sites.

6 Material for preparation

6.1 Questions

- Describe all parts that make up an X-ray tube

- Sketch and describe the X-ray spectrum (photon flux vs. wavelength) of a copper anode without filtering

- What is Cu-Kα_1, Cu-Kα_2 and Cu-Kβ radiation?

- What happens to the spectrum if the radiation is filtered (e.g. by a thin sheet of aluminum)?

- Show, based on Figure 7, that the condition $\vec{d} \cdot \vec{q} = n$ ($n \in \mathbb{Z}$) needs to be met to have maximum constructive interference at a phase difference of ϕ (the scattering vector is defined as $\vec{q} := \vec{k_1} - \vec{k_0}$)

- What is the structure factor?

- What influence does the temperature have on the lattice parameters and the intensities of the diffraction maxima?

6.2 Recommended literature

- Introductory literature

  1. Lecture script "Experimentalphysik V", Prof. R. Kleiner and Prof. F. Schreiber: http://www.soft-matter.uni-tuebingen.de/teaching.html
  2. C. Kittel, Einführung in die Festkörper-Physik, Oldenbourg Verlag, 2002
  3. N.W. Ashcroft, N.D. Mermin, Solid state physics, Harcourt College Publishers

- Advanced literature

  1. J. Als-Nielsen, Elements of modern x-ray physics, Wiley
  2. B.E. Warren, X-ray diffraction, Dover Publications
  3. L. Spieß, R. Schwarzer, H. Behnken, G. Teichert, Moderne Röntgenbeugung, Teubner 2005
  4. A. Taylor, H. Sinclair, On the determination of lattice parameters by the Debye-Scherrer method, Proc. Phys. Soc. **57** (1945) 126