3.17 Strukturanalyse mit Röntgenstrahlen nach Debye-Scherrer

Ausarbeitung (engl.)

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1. Theory

1.1. Very short history of X-rays

X-rays were discovered by Conrad Wilhelm Röntgen in 1895, for which he was honoured with the Nobel Prize in 1905.

The picture on the right shows the first X-ray image, which Conrad Röntgen took of his wife's hand.

He found out that X-rays

- travel in straight lines,
- are exponentially absorbed in matter with the exponent proportional to the mass of the absorbing material
- darken photographic plates
- make shadows of absorbing material on photosensitive paper

First X-ray (1895)

In 1912 Max von Laue assumed some sort of diffraction of X-rays shot on crystals and concluded the wavelength to be around the size of atoms. In the same year William Lawrence Bragg developed Brag's law, which describes the dependence of the angles of maxima of intensity and the distance of the planes of the crystal.
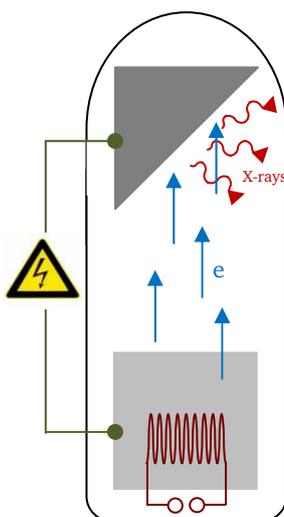
In 1917 Peter Debye and Paul Scherrer developed the method of powder diffraction to analyse the structure of graphite.

This method is described in the following part and was used in our experiment.

1.2. X-rays

"X-rays" is the name for electromagnetic radiation of wavelength in the range of 10^7 to 10^{10} nm. They can be produces in X-ray tubes or synchrotrons.

1.2.1. X-ray tubes

One way to produce X-rays is by X-ray tubes. The heated cathode emits electrons that are accelerated to the anode by high voltage between the electrodes. The efficiency of X-ray tubes is very low. About 99% of the energy is transformed into heat, so the X-ray tube has to be cooled, otherwise the anode would melt. Only about 1% is transformed into X-rays and they do not even go all into the same direction.

The deceleration of the electrons at the anode causes X-rays. There are two kinds of emitted X-rays and each one has a different spectrum.

<u>(I) "Bremsstrahlung"</u>

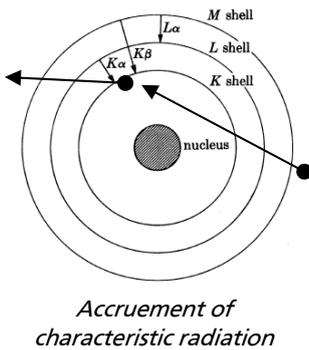Bremsstrahlung is produced directly by the deceleration of the electrons at the anode that is caused by the electromagnetic field of the atoms. If the energy gap between the kinetic energy of the electrons before and after the deceleration is big enough, X-rays are produced. Because of the continuous deceleration of the electrons, the spectrum of these X-rays is continuous, too.

Scheme of an X-ray tube

There is a maximal wavelength λ_{min} (you can see this in the picture on the right, where the spectrums hit the x-axes), that is simply caused by the law of conservation of energy: $h \cdot \nu_{max} = q \cdot U \Longrightarrow \lambda_{min} = \frac{12,4}{U[V]}$ Å. The intensity is proportional to the voltage. If the voltage is high enough, there is another type of X-radiation, the characteristic radiation.
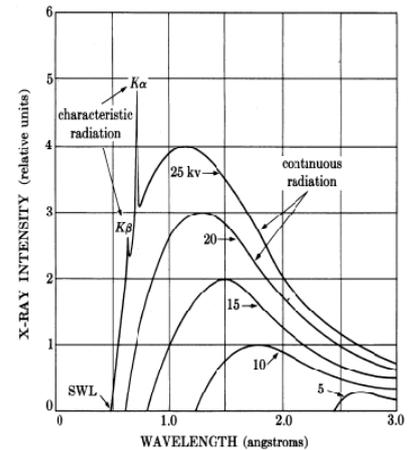

Spectrums of X-rays

(II) Characteristic radiation

If the energy of the accelerated electrons is big enough, the electrons can ionize atoms. They can hit electrons from inner shells of atoms

of the anode. If this happens, the electrons from the inner shell can be kicked out and electrons from outer shells can fill the vacancies to minimize the free energy. The gap between the energy levels is transformed into electromagnetic waves. If the energy gap is big enough, X-rays are produced. Because the energy levels of different materials are not the same, this radiation is characteristic to the material of the anode.

Accruement of characteristic radiation

The energy of the produced X-rays depends on the origin of the vacancy-filling electrons. The different cases are written down with $K_\alpha, K_\beta, L_\alpha$. Because of its higher possibility, the intensity of K_α is 5 to 10 times higher than the intensity of K_β. Also, there are several types of each case. For example, there are six several quantum states for electrons from the L-shell. So, there are six different kinds of K_α-lines. The intensities of $K_{\alpha_{3-6}}$ are negligible, so only K_{α_1} and K_{α_2} are usually discussed. The intensity of K_{α_1} is about twice as high as the intensity of K_{α_2}. That is the reason why you have to weight these lines in different ways to find K_α.

1.2.2. Synchrotrons

X-rays can also be produced in synchrotrons (picture on the right).

In a synchrotron, electrons are first accelerated in a linear accelerator into a booster. There the electrons get relativisticaly accelerated and are led into the storage ring.

To keep the electrons on such a ring, there are bending magnets.

By the acceleration of theses magnets, the synchrotron emits X-rays and other radiation. These are used in laboratories around the synchrotron.

The emitted X-rays are of high brilliance, high polarity and low divergence
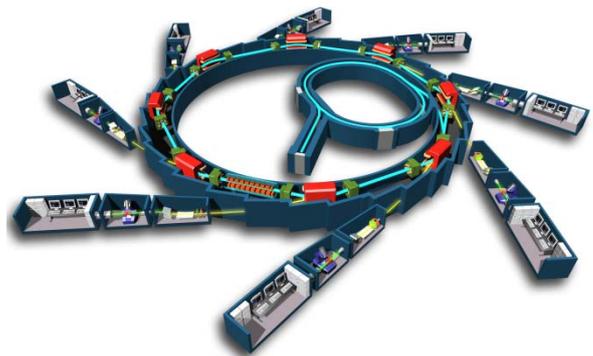
Synchrotron

1.3. Speed of accelerated electrons

In our experiment, we produced X-rays with an X-ray tube. The voltage between cathode and anode was $U = 40\ kV$. With the equation $q \cdot U = \frac{m}{2} v^2$ you can calculate the velocity of the accelerated electrons to about $v \approx \frac{c}{3}$.

1.4. Debye-Scherrer method

The Debye-Scherrer method is a method to analyse polycrystals using X-ray diffraction based on Bragg's law.

The analysed crystal is grinded into a fine powder, because a perfect powder contains all possible crystalline orientations. To support this effect, the sample can be rotated.

Because the experiment is based on Bragg's law and the variation of the angle, there is a need for monochromatic radiation. This is due to the fact, that Bragg's law is a first order equation and so it is only solvable for one variable.

This monochromatic radiation is beamed onto the sample. The sample rotates with a certain angle θ while the detector rotates with the double angle 2θ.

By analysing the diffraction pattern in combination with Bragg's law, you can conclude the distance of the lattice planes and the Miller's indices.
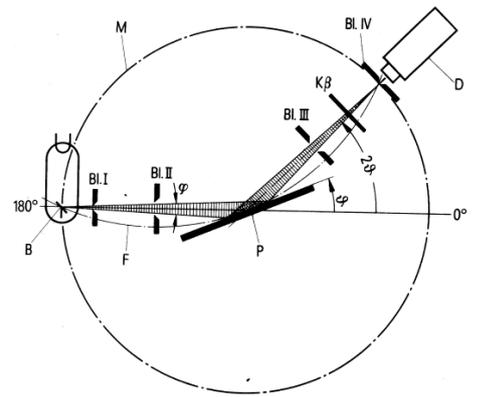
Experimental set-up

Another method was developed by Max von Laue. This method works with a constant angle, but polychromatic radiation on monocrystals. With this method you get bright spots on rings.

1.5. Material of the anode

For experiments on diffraction on atoms, you need wavelength in the size of the atoms. If the wavelength is too high (e.g. UV), Bragg's law is not satisfied: $2d \cdot sin\theta = \lambda \Rightarrow \frac{\lambda}{2d} = sin\theta \leq 1 \Rightarrow \lambda \leq 2d$.

On the other hand, if the wavelength gets too small, it gets harder to resolute the diffraction pattern. Furthermore, the energy of radiation with very small wavelength is very high, so the radiation can penetrate the material without interaction.

The material of our anode was copper (Cu). You often use copper for experiments, because the characteristic radiation (high intensity) is about $K_\alpha \approx 1{,}54$ Å, which is about the size of atoms. So, X-rays from anodes made of copper can be used for experiments on diffraction.

1.6. Bragg's law

If radiation hits a crystal under a certain angle, only a small bandwidth of wavelengths will interfere constructively and a monochromatic beam will leave the crystal.
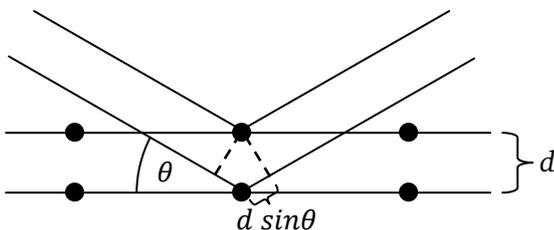
If parallel beams hit different planes, they will have an optical path difference after leaving the crystal. To reach a constructive interference of these out coming beams, this path difference should be a natural multiple of the wavelength:

$$2d\,sin\theta = n\lambda\,;\quad n\epsilon\mathbb{N}$$

The consequence of Bragg's law is a distribution of intensity in dependent on wavelength and angles. In an experimental setup with monochromatic radiation, you will find peaks in a diffractogram, which are caused from the constructive interference under specific angles between the beam and the crystal.

1.7. Filter

The Debye-Scherrer method requires monochromatic radiation. Because the X-ray tube emits non-monochromatic X-rays, we have to use a filter or a monochromating crystal.

(I) Monochromatic Crystal

One possible filter is a monochromatic crystal. It simply uses Bragg's law: $2d\ sin\theta = n\lambda$
The easy the theory is, the hard is the experimental realization. Even a small variation of the angle changes the interval of the out coming monochromatic radiation. So it is very hard to calibrate this filter, especially if you want to modify the equipment.

(II) Absorption filter

Another possible filter is an absorption filter. The loss of intensity of radiation penetrating a material can be described by the following equation:

$$dI = -\mu I\ dx \Rightarrow I = I_0\ e^{-\mu x} = I_0\ e^{-\frac{\mu}{\rho}x\cdot\rho}$$
$$(I: \text{intensity};\ x: \text{distance};\ \rho: \text{density})$$

The so called "mass absorption coefficient" $\frac{\mu}{\rho}$ has a very strong dependence of the wavelength λ and the nuclear number Z.

Experiments showed a dependence of $\frac{\mu}{\rho} \propto \lambda^3 \cdot Z^3$ for every branch. The graph on the right shows this dependence. You also see absorption edges that belong to the different shells.

The reason for those absorption edges is fluorescence. The electrons that hit the crystal do not all ionize it. There are certain energies that equal to the difference of the energies of different possible quantum states of the electrons in the material.

If the wavelength of the accelerated electrons complies with this energy, fluorescence is possible and the absorption increases immediately, as you can see in the graph.

It is obvious, that this method causes a lost of intensity over the whole bandwidth, but it is easy to calibrate.
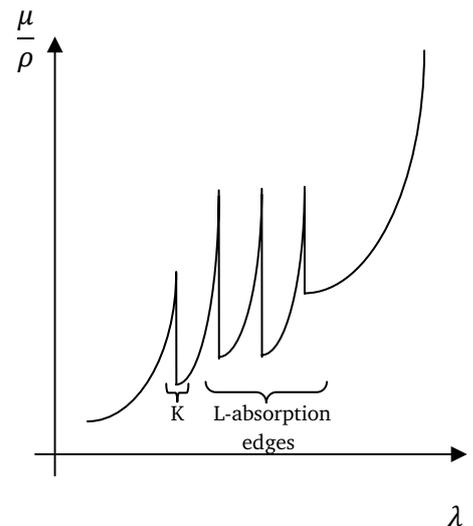
To get the radiation as monochromatic as possible, you can use a material with a certain absorption edge as a filter.

(a) No filter   (b) Nickel filter
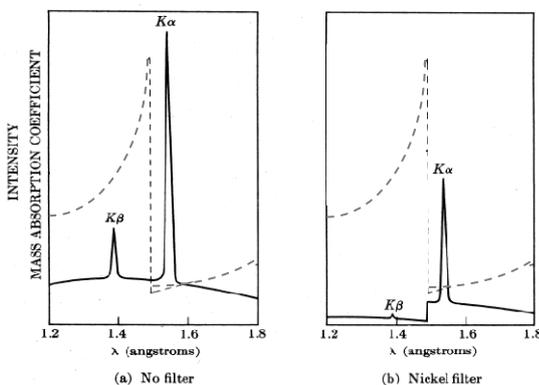
Because of its high intensity, we wanted to detect the K_α-line and reduce the background.

If you look at the graph on the right, you will see that you should use a filter material, whose absorption edge's wavelength is smaller than the K_α-line, but as near as possible. If you compare those wavelengths for several materials, you will see that there is a simple rule: $Z_{filter} = Z_{anode} - 1$. We chose nickel as the material for our filter. ($K_{\alpha\ Cu} \approx 1{,}54\ \text{Å};\ K_{abs\ Ni} \approx 1{,}49\ \text{Å}$)

By a successful filtering, you usually reach ratios of $\frac{I_{K_\alpha}}{I_{K_\beta}} \approx 500$.

1.8. Intensity calculation
The detector only counts the amount of the incoming photons, but not the energy. So it is important to know about all the possible types of scattering to prevent wrong conclusions.

(I) Thomson scattering
The wanted scattering in the Debye-Scherrer method is the Thomson scattering. This coherent scattering describes the electromagnetic interaction between free electrons on photons.
The electromagnetic field of a photon can cause an oscillation of charged particles (e.g. electrons), which induces an electromagnetic field, by which the photon is scattered. This scattering is coherent and elastic and is possible in all directions. Thompson scattering is basis of Bragg's law.
The intensity is given by following equation:

$$I_{Thomson} \sim \frac{I}{r^2 m^2} \left(\frac{1 + cos^2 2\theta}{2} \right)$$

Because of the mass relation between protons and electrons ($m_p \approx 1800 \cdot m_e$) the intensity is negligible for Protons.

(II) Compton scattering
Compton scattering is a non-coherent and inelastic scattering that describes a classical interaction between the electrons by inelastic collisions. The cross-section of Compton scattering compared to Thomson scattering is smaller around energies of keV, but is higher around energies of MeV. So Compton scattering dominates at higher energies and Thomson scattering dominates at lower energies. But in fact, Compton scattering is not negligible for light elements in a normal x-ray.
Because it is non-coherent the intensity will be continuous, so in a diffraction experiment there will be no peaks, but a higher background.

(III) Fluorescence
Fluorescence is another inelastic effect that emits photons. It is an electromagnetic interaction between radiation and matter. If the Energy of a photon is bigger than the bond energy of an electron on e.g. the K-shell, the photon can be absorbed to break the bonding of the electron and the atom. An electron from an outer shell, e.g. the L-shell, will directly replace this lower energy state. Thereby a photon will be emitted, which has the energy of the difference between the two bond energies $h\nu = W_K - W_L$, which is smaller than the energy of the absorbed photon $h\nu \geq W_K$. This emission is non-coherent, too. So there will also be no peaks, but a higher background.

1.9. Miller indices
The Miller indices are a notation system to describe the configuration of crystal lattices. They are defined in the reciprocal lattice:

$$\vec{k}_{hkl} = h\vec{a}^* + k\vec{b}^* + l\vec{c}^*$$

$k_{hkl} = \frac{1}{d_{hkl}}$
$\vec{a}^*, \vec{b}^*, \vec{c}^*$ are the reciprocal lattice vectors with $a^* = \frac{2\pi}{a}, b^* = \frac{2\pi}{b}, c^* = \frac{2\pi}{c}$.

The Miller indices refer to planes in unit cells in the reciprocal lattice.

plan (111)   plan (221)
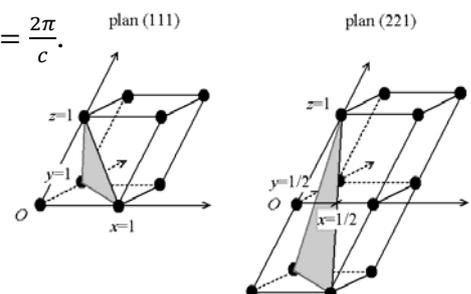
Because of symmetry, the Miller indices (100), (010), (001), ($\bar{1}$00), (0$\bar{1}$0) and (00$\bar{1}$) describe the same configuration, so you usually only talk about (100).

1.10. Intensity of Bragg reflexes

The intensity of the Bragg reflexes is given by following equation:

$$I(h,k,l) = I_0 \cdot |F(h,k,l)|^2 \cdot \rho \cdot \left(\frac{1 + cos^2 2\theta}{2 sin^2 \theta \cdot cos\theta} \right)$$

with the number of equivalent planes ρ.

$F(h,k,l)$ is the structure factor and describes the efficiency of scattering by a unit cell:

$$F(h,k,l) = \sum_r f_r \cdot e^{2\pi i (ha_r + kb_r + lc_r)}$$

f_r is the form factor. This is a correction term referred to point scattering. It describes the ratio between how much is scattered by an atom divided by how much is scattered by an electron, which accords to the efficiency of scattering by an atom.

1.11. Width of Bragg reflexes

The Bragg reflexes have a finite width that is given by the finite size of the atoms and the experimental set-up. In an experiment, especially in an experiment with an x-ray-tube, the light is not perfectly monochromatic and photons are not perfectly parallel. This causes an experimental width of the Bragg reflexes.

2. Homework

2.1. The geometric structure factor for bcc and fcc lattice

The geometric structure factor can be calculated with the formula:

$$S_G = \sum_r e^{2\pi i(h_{u_r} + k_{v_r} + l_{w_r})}$$

The geometric structure factor for an fcc lattice

The coordinates of the atoms for an fcc lattice are $(0,0,0), \left(\frac{1}{2},0,\frac{1}{2}\right), \left(\frac{1}{2},\frac{1}{2},0\right), \left(0,\frac{1}{2},\frac{1}{2}\right)$

If we put these coordinates into the formula we get:

$$S_G = e^0 + e^{i\pi(h+l)} + e^{i\pi(h+k)} + e^{i\pi(k+l)}$$

- If h, k, l are all even or odd we get 4
- If h, k, l are not all even or odd we get 0

For the structure factor F we get:

$$F_{fcc} = \sum_r f_r e^{2\pi i(h_{u_r} + k_{v_r} + l_{w_r})}$$

- If h, k, l are all even or odd we get 4f
- If h, k, l are not all even or odd we get 0

The geometric structure factor for a bcc lattice

The coordinates of the atoms for a bcc lattice are $(0,0,0)$ and $\left(\frac{1}{2},\frac{1}{2},\frac{1}{2}\right)$.

If we put these coordinates into the formula we get:

$$S_G = e^0 + e^{\pi i(h+k+l)}$$

- If h+k+l is even we get 2
- If h+k+l is odd we get 0

For the structure factor F we get:
- If h+k+l is even we get 2f
- If h+k+l is odd we get 0

2.2. How could we use a crystal to monochromatize the x-rays?

The different wavelengths of the x-ray spectrum are scattered in different directions, which is a direct result of Bragg's law. So we have the opportunity to use only one part of the scattered x-rays. To get the best result we can use a monocrystal with a special cut in order to focus photons and therefore preserve the intensity.

2.3. Why should we use the Filter behind the scattering?

If we use the filter after the scattering we could also filter the x-rays which have been changed because of the inelastic scattering (Compton-Scattering, fluorescence). These x-rays could not be used in a helpful way for our experiment.

2.4. Why could we assume that KCl, Cl, K have nearly an equal form factor?

^{19}K will donate one of its electrons to ^{17}Cl: $^{19}K + {}^{17}Cl \rightarrow {}^{18}K^+ + {}^{18}Cl^-$. In that way it will produce an ionic bond. So, both of the atoms will nearly get the same electron configuration. Because of the atomic structure factor the Fourier transformation of the electron density for both form factors will be very similar.

2.5. Which kind of angle is better to analyze the lattice constant more precisely?

If we take the formula (3)

$$sin\vartheta = \frac{\lambda}{2a}n\sqrt{h^2 + k^2 + l^2}$$

and solve that formula for a we get:

$$a = \frac{\lambda}{2sin\vartheta}n\sqrt{h^2 + k^2 + l^2}$$

Now could we show how a is changing with ϑ:

$$\frac{\partial a}{\partial \vartheta} = \frac{\lambda cos\vartheta}{2sin\vartheta}n\sqrt{h^2 + k^2 + l^2} = \frac{a}{tan\vartheta}$$

That means:

$$\frac{\partial a}{a} = \frac{1}{tan\vartheta}\partial\vartheta$$

So we can see that large angles will produce smaller errors then small angles.

2.6. Which kind of angle is better to analyze the grain size more precisely?

If we took the formula

$$\Delta 2\vartheta = 0{,}89\lambda(Bcos\vartheta)^{-1}$$

If we solve that formula for B we can also show the change of B with ϑ:

$$\frac{\partial B}{\partial \vartheta} = \frac{0{,}89\lambda sin\vartheta}{\Delta 2\vartheta cos^2\vartheta} = B\,tan\vartheta$$

That means:

$$\frac{\partial B}{B} = tan\vartheta\partial\vartheta$$

So we can see that smaller angles will produce smaller errors then large angles

3. Experiment

3.1. Analyzing an unknown material

First, we analyzed an unknown material. To identify this material by analyzing its lattice distance, we used the following equation:

$$sin^2\theta = \left(\frac{\lambda}{2a}\right)^2 \cdot (h^2 + k^2 + l^2)$$

$$\Rightarrow a = \frac{\lambda}{2 \cdot \sin\theta} \cdot \sqrt{h^2 + k^2 + l^2}$$

Mr. Fischer gave us the value for $(h^2 + k^2 + l^2)$ of the first angle, where we detected a maximum in intensity. With this value, we could calculate the other values for $(h^2 + k^2 + l^2)$ with the following consideration:

$$\frac{\sin^2\theta_1}{\sin^2\theta_i} = \frac{\left(\frac{\lambda}{2a}\right)^2 \cdot (h_1^2 + k_1^2 + l_1^2)}{\left(\frac{\lambda}{2a}\right)^2 \cdot (h_i^2 + k_i^2 + l_i^2)}$$

$$\Rightarrow (h_i^2 + k_i^2 + l_i^2) = \frac{(h_1^2 + k_1^2 + l_1^2)}{\sin^2\theta_1} \sin^2\theta_i$$

Reflex	2θ	$sin^2\theta$	$h^2 + k^2 + l^2$	(h, k, l)	$(\lambda/2a)$	a (Å)
1	25,840	0,04999265	3	(1,1,1)	0,01666422	5,97195
2	29,894	0,06652553	4	(2,0,0)	0,01663138	5,97784
3	42,743	0,13279728	8	(2,2,0)	0,01659966	5,98355
4	50,568	0,182419	11	(3,1,1)	0,01658355	5,98646
5	53,034	0,1993295	12	(2,2,2)	0,01661079	5,98155
6	61,956	0,26492526	16	(4,0,0)	0,01655783	5,99111
7	68,227	0,31453487	19	(3,3,1)	0,01655447	5,99172
8	70,248	0,33102521	20	(4,2,0)	0,01655126	5,99230
9	78,111	0,39699184	24	(4,2,2)	0,01654133	5,99409
10	83,858	0,44650353	27	(3,3,3)	0,01653717	5,99485

Because the Miller indices for every reflex are either all even or all odd, the structure is face-centered-cubic (fcc). The average of our lattice distance a was 5,98654 Å. With these results, we recognized the unknown material as $AuAl_2$ (literature: a = 5,99730 Å - given by Mr. Fischer).

3.2. Analyzing CsCl

First, we pestled a mixture of silicon and CsCl, we wanted to analyze. To calibrate the gotten peaks of the measurement, we compared our experimental data of silicon with the theoretical one. So, we could determine the angle-shift and the measurement uncertainty. The angle shift is caused by the thickness of the probe. The experimental set-up is calibrated on a specified thickness of the probe. Because the self-made probe will have another thickness than this calibrating one, there will be an angle-shift in the experimental data.
The angle shift is constant over the whole diffractogram. So we can assume:

$$\left(\frac{\Delta 2\vartheta}{2\vartheta}\right)_{Si} = \left(\frac{\Delta 2\vartheta}{2\vartheta}\right)_{CsCl}$$

$$\Rightarrow (\Delta 2\vartheta)_{CsCl} = \left(\frac{\Delta 2\vartheta}{2\vartheta}\right)_{Si} \cdot (2\vartheta)_{CsCl}$$

$\frac{\lambda}{2}\sqrt{h^2 + k^2 + l^2}$

$f(x) = (4{,}118 \pm 0{,}007) \cdot x + (0{,}007 \pm 0{,}003)$

$\sin\theta$
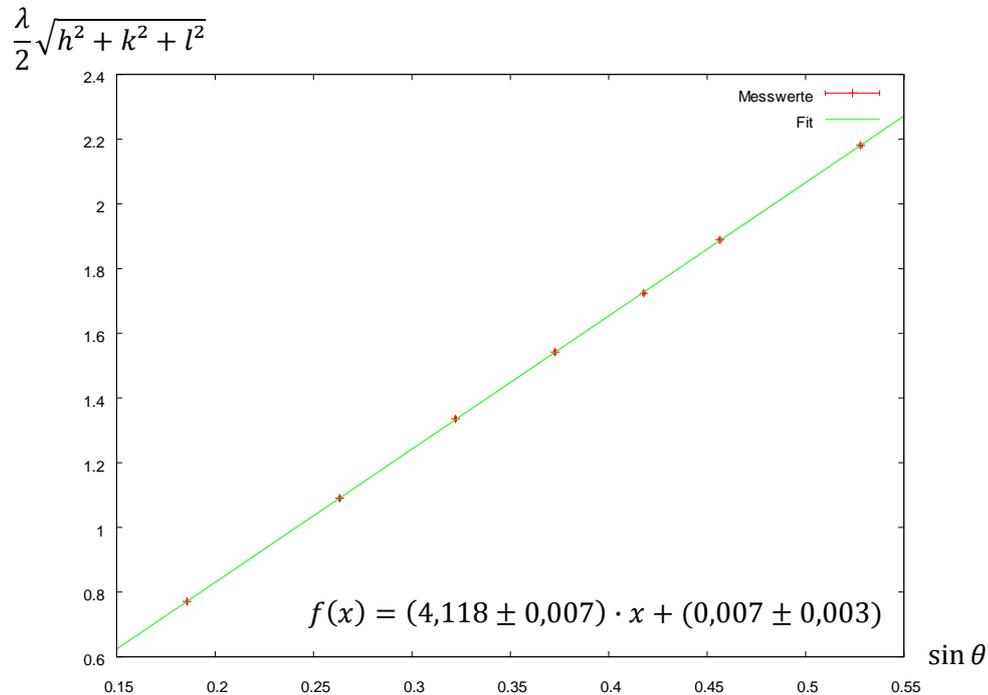
The error $\Delta\sin\vartheta$ is calculated by $\Delta\sin\vartheta = \cos\vartheta \cdot \Delta\vartheta = \cos\vartheta \left(\frac{\Delta 2\vartheta}{2\vartheta}\right)_{Si} \cdot (\vartheta)_{CsCl}$.

The lattice distance a equates to the gradient of the linear function: $a = 4{,}118 \pm 0{,}007$. The literature value $a = 4{,}123$ Å, which was given by Mr. Fischer, is within our error range.

To compare the two methods, we determined the lattice distance with the method of analyzing the unknown material:

Reflex	2θ	$h^2 + k^2 + l^2$	(h, k, l)	a (Å)
1	21,405	1	(1,0,0)	4,15120951
2	30,533	2	(1,1,0)	4,14054943
3	37,586	3	(1,1,1)	4,14487264
4	43,766	4	(2,0,0)	4,13680281
5	49,393	5	(2,1,0)	4,12584857
6	54,328	6	(2,1,1)	4,13624725
7	63,738	8	(2,2,0)	4,12987741
8	68,013	9	(3,0,0)	4,13518856

We calculated the average value for the lattice distance as $a = 4{,}1375$ Å. If you take a look at the determined Miller indices, you will see, that there is a reflex for every possible plane, which indicates the structure to be simple-cubic, which can be confirmed by literature. (Attention! Some sources describe CsCl to be bcc – that is wrong! It would be bcc, if there was only one type of atoms!)

3.3. Analyzing nanocrystalline Pd

To get the grain-size of our material, we used the Debye-Scherrer-formula:

$$\text{FWHM} = \frac{0{,}89 \cdot \lambda}{B \cdot cos\theta}$$

$$\Rightarrow B = \frac{0{,}89 \cdot \lambda}{\text{FWHM} \cdot \cos \theta}$$

If the grain size is very big ($B \gtrsim 1\,\mu m$), the theoretically estimated peak is a Dirac peak. Because the photons are not perfectly monochromatic and parallel, even big grains causes a peak width, which complies with the error of the set-up. To determine this error we first analyzed pure palladium and the corresponding $FWHM_{Pd_{pure}}$ was subtracted to the $FWHM_{Pd_{nano}}$ to get rid of the experimental contribution to the width and to be sure to estimate the grain size correctly.

2θ /°	$FWHM_{Pd_{pure}}$	$FWHM_{Pd_{nano}}$	$\Delta FWHM$	B /Å
40,558	0,264	2,061	1,897	77,12
68,487	0,225	3,090	2,865	57,94

With the literature-value of Pd ($a = 3{,}8908\,\text{Å}$ - given by Mr. Fischer), we concluded our values to be 20 and accordingly 15 unit cells. This way, we estimated an average grain size of 18 unit cells.

4. Final questions

4.1. Why are the reflexes of the K_{a_1} and the K_{a_2} lines further apart for larger scattering angles?

This effect is due to the resolving power $\frac{\lambda}{\Delta\lambda}$ of the experimental set-up. With Bragg's law, you get

$$\lambda = 2d\sin\theta$$
$$\Rightarrow \Delta\lambda = 2d\cos\theta \cdot \Delta\theta$$
$$\frac{\lambda}{\Delta\lambda} = \frac{\tan\theta}{\Delta\theta}$$

If you take a look on the diffractograms, you will see that for large scattering angles, there is a double-peak and for small angles, there is only one wider peak. So you can conclude:

$$\Delta\lambda \approx K_{\alpha_2} - K_{\alpha_1} = 1{,}54439\,\text{Å} - 1{,}540562\,\text{Å} = 0{,}003828\,\text{Å}$$

With this value, you can calculate the reflex-distance as:

$$\Delta\theta = \left(\frac{\lambda}{\Delta\lambda}\right)^{-1}\tan\theta \approx 0{,}0025 \cdot \tan\theta$$

So, for small angles, the reflexes are closer and for great angles, they are further apart.

4.2. Are x-rays suitable for studying compounds which consist of very different atomic numbers?

No, because the Interferogramm depends strongly on the atomic form factor the element with the larger atomic number would dominate the atomic form factor. This way we wouldn't learn much about the other part of the compound.

4.3. What is a "forbidden" reflex?

Bragg's law is based on a simple crystal structure with only one atom as the basis. If there is a basis with a more complicated structure, the diffraction pattern will look different. This aberration is given by the structure factor. It is possible, that this causes an intensity of $I = 0$, although Bragg's law is fulfilled and there should be constructive interference. These reflexes, for which $F_{hkl} = 0$, are called "forbidden".

4.4. Is it possible to analyze a metallic copper-sample using a copper anode?

There would only be a problem to analyze a material if it would absorb parts of the monochromatic radiation and emit it at random directions (fluorescence) without changing its wavelength.
As written in the theoretical introduction, the wavelength is changed: If the Energy of a photon is bigger than the bond energy of an electron on e.g. the K-shell, the photon can be absorbed to break the bonding of the electron and the atom. An electron from an outer shell, e.g. the L-shell, will directly replace this lower energy state. Thereby a photon will be emitted, which has the energy of the difference between the two bond energies $h\nu = W_K - W_L$, which is smaller than the energy of the absorbed photon $h\nu \geq W_K$.
Because $\lambda_{K_\alpha} \approx 1{,}54\,\text{Å} \neq \lambda_{K_{absorption\,edge}} \approx 1{,}38\,\text{Å}$, there is not enough energy for ionizing the K-shells of the copper-sample. So, the absorption is not too big to disturb the experiment.

4.5. The grainsize of a sample material is usually not the same for all the grains. The Scherer- Formula is therefore only an approximation for the different grain sizes. How are the different sizes taken into account?

The Sherrer formula $\text{FWHM} = \frac{0,89 \cdot \lambda}{B \cdot cos\theta}$ is based on the size of the crystallites and the amount of the "out of phaseness" they can withstand.

If the grain size is very big ($B \gtrsim 1\,\mu m$), the theoretically estimated peak is a Dirac peak. Because the photons are not perfectly monochromatic and parallel, even big grains causes a peak width, which complies with the error of the set-up. This error is constant for all grainsizes, so the approximation will be more exactly for small grainsizes, because big grains have no contribution and small grains a lot.

4.6. One of the questions of the preparing tasks dealt with inelastic scattering by creation or annihilation of phonons. Describe the effects on the diffractogram. Which physical quantity describes these processes?

In our model the atoms are ordered in plains with no thickness, this doesn't hold true in reality.
The physical quantity which is responsible for a thickness is the Temperature.
Thermal energy leads to vibrations of the atoms around their equilibrium position (phonons). This causes a thickness of the idealized planes.
One effect that is due to the creation and annihilation of phonons is the blurring of constructive interference that leads to a decrease in intensity of the peaks.
Another effect that is due to the same origin is Thompson scattering in all directions, which causes an increasing background. This effect gets stronger, the "softer" the analyzed crystals are and the lower their boiling point is.

5. Sources

5.1. Literature

Manual to the experiment
http://epswww.unm.edu/xrd/xrdclass/03-GenX-rays.pdf
http://en.wikipedia.org/wiki/X-ray_crystallography#Early_scientific_history_of_crystals_and_X-rays
http://en.wikipedia.org/wiki/Powder_diffraction

5.2. Pictures

First X-ray:	http://www.nlm.nih.gov/dreamanatomy/images/400%20dpi/Z1.jpg
Spectrum of X-rays:	Manual to the experiment
Accruement of characteristic radiation:	Manual to the experiment
Synchrotron:	http://upload.wikimedia.org/wikipedia/commons/6/60/Sch%C3%A9ma_de_principe_du_synchrotron.jpg
Experimental set-up:	Manual to the experiment
Absorption filter intensity:	Manual to the experiment
Miller indices:	http://upload.wikimedia.org/wikipedia/commons/e/e8/Indices_miller_plan_definition.png