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guide for the exercise



within the course 511.121 Praktikum für Fortgeschrittene (511.121 Advanced Laboratory Exercises)

and for the exercise

# **Applied X-ray diffraction**

within the course 513.119 Experimentelles Praktikum (513.119 Experimental Laboratory Exercises)



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#### Preface

This script was written by Martin Oehzelt and Thomas Haber as a guide for the exercise X-ray Diffraction within the course Praktikum für Fortgeschrittene (511.121). The contents are based on the diploma thesis of T. Haber [1]. This manuscript is also suited for the preparation regarding the exercise Applied X-ray Diffraction within the course Experimentelles Praktikum (513.119).

The aim of the course is to learn to apply the theoretical basics of xray diffraction to real problems, to get operating experience with the lab equipment and to strengthen the skills in data evaluation. Therefore a certain level of preparation is indispensable. Please read through the script before attending the course. The knowledge of each participant will be checked at the beginning of the exercise. The preparation for the course contributes one third of the achievable points. Please note that no formulas except of *Bragg's law* will be asked. The most important topics are:

- X-ray radiation: Bremsstrahlung and characteristic radiation; generation and detection
- what is a crystal lattice, a unit cell and a lattice plane
- The principle of spherical and stereographic projection
- Two conditions for diffraction: Bragg's law and parallelism condition
- Powder Diffraction ( $\theta/2\theta$ -scans with the SIEMENS)
- Single Crystal Diffraction (Pole Figure Measurements with the PHILIPS)

When writing the lab log please add your name, matriculation number, email address as well as the date of the exercise.

Please help to improve the script by pointing out mistakes and flaws, so that they can be corrected. Thanks in advance, Thomas Haber.

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## Chapter 1

## **Fundamentals**

## 1.1 X-Ray Radiation

X-rays are electromagnetic waves with a wavelength in the range of  $10^{-5}$  to 100 Å. This wavelength is generated by energy-loss of electrons. There are two different ways for electrons to lose their energy, depending whether they are free or bound. If they are free they can interact with any material (e.g. elastic scattering, inelastic scattering) and lose an arbitrary amount of their kinetic energy which leads to a continuous spectrum (section 1.1.1). In case of bound electrons they lose a certain amount of their potential energy when they change their energy level. Since these electronic levels have discrete energies, X-rays with certain wavelengths are generated. The energy levels and thus the wavelengths are naturally dependent on the material and therefore the spectrum of the X-rays are characteristic for a material (section 1.1.2).

#### 1.1.1 Bremsstrahlung

The continuous radiation is emitted when electrons accelerate or decelerate (e.g. in the field of an atomic nucleus). The wavelength of the emitted X-ray depends on the energy loss or gain of the electron. Therewith there is a minimum wavelength  $\lambda_{min}$  corresponding to the full kinetic energy of the electron  $E_{max}$ . The kinetic energy is the electron charge e times the acceleration voltage V which leads to

$$E_{max} = eV = h\nu_{max} = \frac{hc}{\lambda_{min}} \tag{1.1}$$

$$\lambda_{min} = \frac{hc}{eV} \tag{1.2}$$



Figure 1.1: a) shows some schematic spectra of Bremsstrahlung dependent on the acceleration voltage and b) shows the spectra with the additional characteristic features.

where  $e = 1.602 \cdot 10^{-19} C$  is the charge of the electron,  $h = 6.6255 \cdot 10^{-34} Js$  is Planck's constant and  $c = 2.2998 \cdot 10^8 m s^{-1}$  is the light speed.

This is the reason for a cut off at the lower end of  $\lambda_{min}$ . The probability to lose  $E_{max}$  at once is rather low. Every single interaction leads to a deceleration and to an X-ray photon. The average over many electron-atom interactions results in the continuous spectrum. These typical spectra are shown in Fig. 1.1(a). The continuous X-ray spectrum is used e.g. for X-ray radiography in medicine where typically tungsten X-ray tubes are used.

#### 1.1.2 Characteristic X-Ray Radiation

Additionally to the Bremsstrahlung a material dependent characteristic Xray radiation can occur. When a material is hit by high energy electrons there is a probability to ionize these atoms. The emitted electrons had a well defined binding energy and electron from higher electron levels (less binding energy) will relax to the unoccupied electronic states. When an electron changes its energy level, an X-ray photon is emitted. The energy of this photon is given by the difference of the binding energies of the two levels. Since the energy levels depend on the material the energies of the emitted X-ray photons are characteristic for each atom.

Within an atom there are several shells of energy levels (K-, L-, M-, ...) and an emitted electron can be replaced by an electron of any higher shell. The nomenclature of the resulting wavelengths is related to the shells which

are involved in the transition. The first letter is always the refilled shell. The second letter is denoted in small Greek letters increasing with the distance of the shells. The electronic transition between shells  $L \to K$  emits the  $K\alpha$  radiation whereas  $K\beta$  is emitted by the transition  $M \to K$  and  $L\alpha$  by  $M \to L$ . Due to the spin-orbit coupling several energy levels are possible within one shell. There are for example three different levels in the L shell. Two of them allow an  $L \to K$  transitions ( $L_{II}$  and  $L_{III}$ ). These transitions lead to  $K\alpha_1$  and  $K\alpha_2$ . Indeed their splitting is very small and in many cases the last index is neglected concerning experiments. A schematic spectrum of the characteristic radiation additionally to the continuous spectrum is shown in Figure 1.1(b).

### 1.1.3 X-ray Generation



(a) Philips Glass Diffraction X-Ray Tube.



(b) Open view of the tube.

Figure 1.2: A typical X-ray tube used in X-ray diffractometers. In Figure a you can see the tubular metal body with the Beryllium window. Figure B shows the inside of the tube where you can see the water nozzle for cooling. Of course a tube cannot be opened because of vacuum conditions.

Conventional X-ray tubes are vacuum tube diodes with a cathode made of Tungsten and used up to -50kV. The electrons are emitted and accelerated to the anode. There the accelerated free electrons hit the electron cloud of the anode and are decelerated. The anode material is partly ionized and inner



Figure 1.3: Schematic drawing of a synchrotron with the storage ring and the beam lines.

shell electrons are emitted. This state of the anode atom is not stable and relaxes when electrons of higher levels fill up the empty inner shell states. In this process X-ray radiation is generated. The energies of the different states depend on the material as well as the wavelengths of the generated X-rays.

The X-rays are emitted through Beryllium windows which are shown in Figure 1.2(a). Of course the radiation is generated in all directions which means that only the minor part of the totally generated radiation really transmits the tube windows. The main part is absorbed and converted into heat. The cooling is very important to run the X-ray tube which is shown in Figure 1.2(b) and realized by a cold water flow through the tube.

A different way to generate photons with the wavelength of X-rays is used in synchrotrons. In X-ray tubes the accelerated electrons are decelerated when hitting a material. The electrons in a synchrotron are running in an UHV (ultra high vacuum) storage ring. There the electrons have a constant velocity near c. As every accelerated charge generates an electromagnetic wave and the electrons in the storage ring are forced to a constant orbit, these electrons experience centrifugal forces. This centrifugal forces accelerate the electrons in a new direction and are the origin of the so called synchrotron radiation. If the velocity of the electrons and the centrifugal force have a certain value, synchrotron radiation with a wavelength of X-rays is generated. The intensity of a synchrotron is by several orders of magnitude higher. Additionally the radiation of a synchrotron is much higher collimated than the X-rays from a tube. Furthermore the wavelength of a synchrotron is tuneable, which is very useful for numerous experiments. Figure 1.3(a) shows a schematic drawing of a synchrotron with the storage ring and the different



Figure 1.4: Two of the most used detectors for X-ray radiation. A gas filled proportional counter (a) for large count rates of ionization and a solid state detector for energy dispersive detection (b).

beam lines.

## **1.2 X-Ray Detectors**

The principle of X-ray detection is to convert high energy photons to electric signals. One possibility to realize it is to use a *gas-filled proportional detector* which is shown in Figure 1.4(a). The detector is filled with an inert gas and an X-ray photon ionizes the gas. Between the two electrodes high voltage is applied which accelerates the charged molecules to one of the electrodes. On its way through the gas the ions can ionize other atoms and the current measured is proportional to energy of the incoming photon. The numbers of photons are counted. The energy dispersion is usually rather weak.

Another detector is the *scintillator*. It consists of an optically active metal, usually NaI, that generates a brief optical flash when an X-ray photon is absorbed. This light is transferred to a photomultiplier tube whose photocathode emits electrons when illuminated. The flash does not depend on the energy of the X-ray photon and thus detection is not energy dispersive.

The third kind of X-ray detectors are **solid state detectors** as shown in Figure 1.4(b). They consist of a Si or Ge diode operated with reverse bias. An incoming X-ray photon causes additional charge carriers which lead to a change of current that is detected. The quantity of the charge carriers is dependent on the energy of the X-rays which enables an energy dispersive measurement. Both the count rate and the resolutions of a solid state detector is comparably low to the other detectors. However they enable an energy dispersive measurement and are thus indispensable for numerous applications.

## 1.3 Crystallography

This section is just be a very brief introduction to crystallography pointing out the most important things concerning this exercise: the crystal lattice, its unit cell, the basic nomenclature in crystallography, and the concepts of diffraction. For more details books of solid state physics or dedicated books for crystallography can be recommended. [2]

### 1.3.1 Crystal Lattice and the Unit Cell

The lattice of a crystal is described by a right handed coordinate system which is not necessarily orthogonal. Since this coordinate system defines the *lattice* of the crystal, the length of the base vectors are called lattice constants  $|\vec{a}|, |\vec{b}|$  and  $|\vec{c}|$  as well as the angles  $\alpha$ ,  $\beta$ , and  $\gamma$  between them. A parallelepiped is formed by the lattice constants, which is called the *unit cell* of the crystal. Any lattice point is characterized by the lattice vector  $\vec{r} = u\vec{a} + v\vec{b} + w\vec{c}$  where u,v and w are integers. It means that starting from any point  $\vec{r}'$  the crystal is completely equivalent at a point  $\vec{r}''$  when  $\vec{r}''$ :

$$\vec{r}'' = \vec{r}' + u\vec{a} + v\vec{b} + w\vec{c} \tag{1.3}$$

In some simple crystals the corners of the unit cell are equivalent to the position of the atoms which is not the case in most of the crystals. In other words the lattice describes the periodicity of the crystal but not the position of atoms or molecules. The lattices can be classified by seven *crystal systems* listed in Table 1.1 and 14 *Bravais–lattices* shown in Figure 1.5 ([3, p. 32] and [2, p. 66]).

crystal system	lattice constants	angles
triclinic	$a \neq b \neq c \neq a$	$\alpha \neq \beta \neq \gamma \neq \alpha$
monoclinic	$a \neq b \neq c \neq a$	$\alpha = \gamma = \frac{\pi}{2}$
orthorombic	$a \neq b \neq c \neq a$	$\alpha = \beta = \gamma = \frac{\pi}{2}$
tetragonal	$a = b \neq c$	$\alpha = \beta = \gamma = \frac{\pi}{2}$
rhombohedral	a = b = c	$90 \neq \alpha = \beta = \gamma < \frac{2\pi}{3}$
hexagonal	$a = b \neq c$	$\alpha = \beta = \frac{\pi}{2}; \gamma = \frac{2\pi}{3}$
cubic	a = b = c	$\alpha = \beta = \gamma = \frac{\pi}{2}$

Table 1.1: Lattice constants and angles of the crystal systems.

#### **1.3.2** Lattice Planes

A plane passing through three lattice points is called *lattice plane*. In principle it is possible to describe any plane by its axis intersections as an integer multiple of  $|\vec{a}|$ ,  $|\vec{b}|$ ,  $|\vec{c}|$  as shown in Figure 1.6 where the plane could be named (324). In case of planes parallel to one or two base vectors with no intersection between them (except at  $\infty$ ) this nomenclature becomes unhandy. It is more convenient to take the reciprocal values of the intersections, multiplied with the least common multiple, to name the planes. The plane in Figure 1.6, according to the reciprocal values  $(\frac{1}{3}\frac{1}{2}\frac{1}{4})$ , would be denoted as (463) plane. The problem mentioned above is thus solved because no intersection lead to the value of  $\infty^{-1}$  which is zero. These indices (hkl) are called *Miller Indices* of a plane. Due to the equivalence of all lattice points the (hkl)-plane is not a single plane but the entirety of all equivalent planes in the crystal parallel to each other.

## 1.4 The Spherical and Stereographic Projection

The spherical projection is the first step to a representation of a three dimensional lattice. As shown in Figure 1.7 the idea is to draw a unit sphere around the crystal and project its plane normal vectors onto this sphere. In Figure 1.7 the projected planes are limited to *Miller indices* lower than two. The result is a three dimensional unambiguous projection of the crystal structure. The intersections are called *poles* and are denoted with the indices



Figure 1.5: Unit cells of the 14 Bravais-lattices: 1: triclinic; 2, 3: monoclinic; 4, 5, 6, 7: orthorombic; 8: hexagonal; 9: rhombohedral; 10, 11: tetragonal; 12, 13, 14: cubic.



Figure 1.6: The lattice plane  $(hkl) = (4 \ 6 \ 3)$ .

of the plane. To determine a pole by a tuple  $(\varphi, \psi)$  one lattice plane is chosen to be at  $(\varphi, \psi) = (0, 0)$ , in this case (100) and a second plane, in this case (010) is chosen which is also at  $\psi = 0^{\circ}$  and  $\varphi = 90^{\circ}$ . The lines to the poles span a plane, which is in this case the x-y-plane as shown in Figure 1.7. This way each pole has a well defined tuple  $(\varphi, \psi)$  with  $\varphi$  measured clockwise in the range of  $0...360^{\circ}$  and  $\psi$  is the angle to the x-y-plane in the range of  $-90...90^{\circ}$ .

In general a three dimensional projection is not very useful because most illustrations are printed and therefore limited to two dimensions. The stereographic projection is a way to project the poles of the spherical projection to a two dimensional plane. Figure 1.8 shows the principle of the stereographic projection where P represents a point of the spherical projection. A line to the south pole (or the north pole if the point is in the lower hemisphere) is drawn which intersects the plane of  $\vec{e_1}$  and  $\vec{e_2}$  at the point P'. The result is a two dimensional picture of the poles and thus a two dimensional representation of the crystal structure. The tuple ( $\varphi, \psi$ ) can be calculated from the position of P'. The angle  $\varphi$  of course remains the same in the spherical projection and the angle  $\psi$  can be calculated by:

$$r' = \tan\frac{\psi}{2} \tag{1.4}$$

To make the stereographic projection easier to read, additionally to the poles a polar net is projected in the same way, which is shown in Figure



Figure 1.7: The spherical projection of a crystal structure is the projection of the planes on a unit sphere around the crystal. In this case no higher *Miller indices* than one are used. The projection of a plane is realized by the intersection of its plane normal vector and the unit sphere. This leads to an unambiguous three dimensional projection of the crystal structure.

1.9. The polar net is drawn on the unit sphere with circles in steps of  $10^{\circ}$  and lines every  $45^{\circ}$  in  $\varphi$ . On the right side of the picture the stereographic projection of the net is shown which makes a grid for the tuples  $(\varphi, \psi)$ .

### 1.5 Diffraction

In a diffraction experiment an incident beam is directed to the specimen and diffracted intensity is detected. Diffraction of electromagnetic waves is described within the theory of electrodynamics. A mathematical approach is shown in [4, p. 28-32]. To understand the methods used within this lab it is enough to mention the most important conditions of diffraction. The incident beam is named  $\vec{k}_0$  and the diffracted beam is  $\vec{k}$  while the difference of these vectors, as shown in Figure 1.10, is called **scattering vector**  $\vec{S}$  and



Figure 1.8: The stereographic projection is a projection of the poles, generated with the spherical projection, onto the plane of  $\psi = 0$ . The point P' is located at the intersection of the plane and a line from the point P to the 'south pole'.

is defined as:

$$\vec{S} = \vec{k} - \vec{k}_0 \tag{1.5}$$

### 1.5.1 Bragg's Law

A very simple approach that derives *Bragg's law*, can be given by geometrical consideration of the problem. As seen in Figure 1.10 the incident beam hits the sample with an angle  $\theta$  between the beam and a certain lattice plane. The beam is diffracted by the planes whereby the lower part has a longer optically path. To get constructive interference the additional way s has to be an integer multiple of  $\lambda$ . The way can be easily calculated with geometrical considerations to be  $s = 2d \sin \theta$ . Since this has to equal the wavelength or an integer multiple of it the result is:

$$n\lambda = 2d_{hkl}\sin(\frac{2\theta}{2}) \tag{1.6}$$

This equation is the so called Bragg's law and it is the **first condition** of



Figure 1.9: The polar net drawn on the unit sphere with circles of latitude drawn every 10° and lines of longitude every 45° and its stereographic projection on the right side. As you can see the spaces between the circles of the projection increase with increasing  $\psi$ .

diffraction. The term  $2\theta/2$  instead of  $\theta$  is chosen because  $2\theta$  is the important diffraction angle. The incident angle  $\theta$  can be changed without variation of Bragg's law (see section 1.6.4). The **second condition** is the parallelism condition which says that the scattering vector  $\vec{S}$  has to be parallel to the plane normal vector or perpendicular to the plane. In other words the incident angle  $\theta$  has to equal the outgoing angle. It is to say that the angles are measured between the beams and the net planes and not between the beams and the surface! The complete derivation of these two conditions is based on the **Laue conditions** that say that the scattering vector has to equal the plane normal vector [1, p.16-20].

## **1.6** Experimental

### 1.6.1 X-Ray Methods

Two different X-ray diffractometers will be used within this lab course. A Siemens D501 Kristalloflex powder diffractometer (coupled  $\theta/2\theta$ ) (Figure 1.11) and a four-circle-texture-goniometer Philips X'PERT, equipped with an ATC3 cradle (Figure 1.14(b)). The Siemens system works with a copper tube while the Philips goniometer is used with a chromium tube. The wavelengths of these tubes are listed in Table 1.2.



Figure 1.10: The incident beam  $\vec{k}_0$  is diffracted on the lattice plane  $d_{hkl}$ . The diffracted beam is  $\vec{k}$ . The scattering vector  $\vec{S}$  is defined by equation 1.5. To get positive interference *Bragg's law* (equ. 1.6) has to be fulfilled.

Table 1.2: The systems, the radiation sources, the according wavelengths and their percentage after the monochromator.

system	radiation	wavelength / Å	percentage / $\%$
Siemens	$CuK\alpha$	1.54	> 99.9
	$CuK\beta$	1.39	< 0.1
Philips	$CrK\alpha$	2.29	> 99.9
	$CrK\beta$	2.08	< 0.1

### 1.6.2 The X-ray Powder Diffractometer

The X-ray powder diffractometer (coupled  $\theta/2\theta$ ) shown in Figure 1.11 is the most common application for X-ray diffraction. The image shows a Siemens D501 Kristalloflex powder diffractometer which is used during this lab. The X-ray beam leaves the X-ray tube through the slits one and two and hits the sample in the center of the goniometer. The angle between the incident beam and the surface of the sample is  $\theta$ . The detector is on the right hand side with a monochromator in front which has an angle of  $2\theta$  with respect to



Figure 1.11: A Siemens D501 Kristalloflex powder diffractometer (coupled  $\theta/2\theta$ ): The beam is generated in the X-ray tube (I) which is mounted at the left side. The sample (II) is rotating in the center with an angular speed of  $\omega$ . The beam is diffracted and passes the monochromator (III) in front of the detector (IV). The monochromator and the detector have the angular speed  $2\omega$ .

the incident beam. The diffracted beam passes the slits three and four before being detected. When measuring the diffracted beams the detector is turned with the double angular velocity as the sample, so that the angle between the incoming beam and the surface equals the angle between the diffracted beam and the surface. This arrangement is called *Bragg Brentano Focusing*.

### **1.6.3** X-Ray $\Theta/2\Theta$ Measurements

As mentioned in section 1.5.1 Bragg's law has to be fulfilled to get constructive interference which results in detectable diffracted intensity. Additionally this equation only holds if the incident beam and the diffracted beam are symmetric with respect to the measured planes. In case of randomly oriented crystalline powder there are always planes with this symmetric orientation present. Since the used X-ray wavelength is defined by the X-ray tube and additionally selected with a monochromator there are only two of the three variables in Bragg's law (equ. 1.6) tunable and dependent on each other: the net plane distance  $d_{hkl}$  and the scattering angle  $2\theta$ . Experimentally changeable is only the angle  $2\theta$ . When rotating the detector with the doubled angular speed as the specimen you will detect intensities at certain positions  $(2\theta)$  which are assigned to certain net plane distances  $d_{hkl}$  and thus to certain net planes (hkl). This measurement is called  $\theta/2\theta$ -measurement and an example is shown in Figure 1.12. In these  $\theta/2\theta$ -scans all detected planes are parallel to each other, but the net plane distances are different. The intensities of the peaks depend on several intensity factors such as the structure factor or instrumental influences. In general there are only few reflections that have detectable intensities.



Figure 1.12: Two powder patterns calculated with the program *Powder Cell* of para-sexiphenyl (blue) and KCl (red). All peaks measured at a certain angle  $2\theta$  correspond to a certain net plane distance  $d_{hkl}$  and can thus be assigned to a certain net plane (*hkl*).

In case of a single crystal the second condition will be fulfilled only for net planes which are parallel to the surface. Making a  $\theta/2\theta$  measurement on a single crystal will thus detect nothing but the plane which is parallel to the surface.

#### 1.6.4 X-Ray Rocking Curve Measurements



Figure 1.13: A Rocking Curve is a measurement to determine the quality of the alignment of crystallites (mocaicity).

When performing a Rocking Curve the detector is fixed, and thus the diffraction angle  $2\theta$  is constant. Regarding Bragg's law only one certain net plane distance is chosen by fixing the diffraction angle. A Rocking Curve is usually performed at a found peak of a  $\theta/2\theta$ -scan to check the spatial distribution of the crystallites. This is realized by tilting the sample, in other words the incident angle. Therefore a Rocking Curve probes the parallelism condition  $(\vec{S} \parallel \vec{n})$ . As shown in Figure 1.13 a Rocking Curve probes the quality of the alignment of the crystallites. If they are distributed completely randomly (powder) a Rocking Curve won't lead to a peak but rather yield an almost constant intensity. The better the crystallites are aligned the better the Rocking Curve will be. The best Rocking Curves are obtained when investigating a single crystal since there is only one crystal.

#### 1.6.5 X-Ray Pole Figure Technique

The X-Ray Pole Figure Technique is one of the most powerful measurements for determining the entirety of crystal orientations and their alignment relative to each other. The principle of making a pole figure is shown in Figure 1.14. The measurement is done at a certain  $2\theta$  angle so that Bragg's law is always fulfilled for a chosen net plane. During the measurement the sample is rotated and tilted to probe all possible  $\varphi$  and  $\psi$  angles in the upper hemisphere of the sample. This is done by a rotation of the sample from  $\varphi = 0^{\circ}$ to 360° followed by a certain tilt of  $\Delta \psi$  and another rotation. Tilting the sample from  $\psi = 0^{\circ}$  to 90° with a  $\varphi$  rotation for each step, the whole upper hemisphere is probed which leads to a stereographic projection as shown in Figure 1.8. The big difference to the stereographic projection is that a pole figure is performed at a fixed diffraction angle  $2\theta$  and thus only net planes with the same net plane distance  $d_{hkl}$  are detected. A peak in a pole figure



(a) The principle of making pole figures



(b) A Philips X'Pert four-circle-texture-goniometer

Figure 1.14: The principle of X-Ray Pole Figure Technique: the measurement is done at a fixed  $2\theta$  angle so that Bragg's law is always fulfilled. The sample is rotated around  $\varphi$  and  $\psi$  so that every net plane's normal vector  $\vec{n}$  is once parallel to the scattering vector  $\vec{S}$ . The intensities are mapped by stereographic projection according to their tuple  $(\varphi, \psi)$ . The measurement are done at a Philips X'Pert four-circle-texture-goniometer (b). is called enhanced pole density (EPD) and corresponds to a certain pole and therewith to a certain net plane. Poles with a different net plane distance can be measured within another pole figure using a different  $2\theta$  angle according to the selected net plane.

### 1.6.6 Silicon

Silicon (Si) crystals of high purity can be manifactured and thus the crystal structure is well defined and close to a perfect crystal structure. For this reason Si is often used to align X-ray diffractometers. In this lab course the *Standard Reference Material 640c – Silicon Powder NIST* and a silicon single crystal will be used<sup>1</sup>. The silicon crystal has a cubic crystal structure with lattice constants of:

$$a = 0.54311946nm \pm 0.00000092nm \tag{1.7}$$

The diffraction angles for  $Cu_{K\alpha}$  and  $Cr_{K\alpha}$  radiation for various net plane distances are listed in Table 1.3 using Braggs's law (equ. 1.6). The spatial angles of net planes relative to each other are denoted for the most important planes for a cubic system in Table 1.4.

 $<sup>^1\</sup>mathrm{If}$  other materials will be used the necessary documents will be delivered during the exercise

h	k	l	$d_{hkl}$	$2\theta_{Cu}$	$2\theta_{Cr}$
1	1	1	3.14	28.44	42.83
2	2	0	1.92	47.30	73.20
3	1	1	1.64	56.12	88.71
4	0	0	1.36	69.13	114.96
3	3	1	1.25	76.37	133.51
4	2	2	1.11	88.02	
5	1	1	1.05	94.95	
4	4	0	0.96	106.70	
5	3	1	0.92	114.08	
6	2	0	0.86	127.53	
5	3	3	0.83	136.88	

Table 1.3: Net plane distances and according diffraction angles for Silicon diffractions of  $Cu_{K\alpha}$  ( $\lambda = 1.5406\text{\AA}$ ) and  $Cr_{K\alpha}$  ( $\lambda = 2.28975\text{\AA}$ ) radiation. Since this is a cubic system the values are equivalent for ( $\pm h \pm k \pm l$ ).

Table 1.4: Angle between net planes relative to each other in real space  $(\psi)$  for a cubic crystal.

plane 1 $\{hkl\}$	plane 2 $\{hkl\}$	$\psi/^{\circ}$
{100}	$\{100\}$	90
{100}	$\{110\}$	45
{100}	{111}	54.7
{100}	{311}	25.2; 72.5
{111}	{100}	54.7
{111}	{110}	35.3;90
{111}	{111}	70.5
{111}	{311}	29.5; 58.5; 80

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## Chapter 2

## The Exercise

Date

The samples and problems will be discussed during the exercise.

## 2.1 Samples to investigate

- Sample 1: ...
- Sample 2: ...
- Sample 3: ...

## 2.2 The Problem

- 1. Mount the sample and set the correct settings.
- 2. Perform a rough  $\theta/2\theta$ -scan ...
- 3. . . .