Debye-Scherrer diffraction patterns with a cubic powder sample

Physics > Modern Physics > Production & use of X-rays

Difficulty level	Group size	Preparation time	Execution time
hard	2	45+ minutes	45+ minutes

This content can also be found online at:

http://localhost:1337/c/5f775b76ca39fd0003291be8

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General information

Application

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Setup

Most applications of X rays are based on their ability to pass through matter. Since this ability is dependent on the density of the matter, imaging of the interior of objects and even peaple becomes possible. This has wide usage in fields such as medicine or security.

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Other information (1/2)

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Prior knowledge

The prior knowledge required for this experiment is found in the Theory section.

Main principle

A cubic crystalline powder sample is irradiated with the radiation from a X-ray tube with a copper anode. A Geiger-Mueller counter tube is automatically swivelled to detect the radiation that is constructively reflected from the various lattice planes of the crystallites. The Bragg diagram is automatically recorded. A graphical evaluation procedure is used to assign the reflexes to the individual lattice planes and to determine the corresponding Bravais lattice type as well as the lattice constant of the substance.

Other information (2/2)

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Learning objective

The goal of this experiment is to get to investigate Debeye-Scherrer patterns for Bragg-Brentano-geometry.

Tasks

1. Record the intensity of the Cu X-rays back scattered by a cubic powder sample as a function of the back scattering angle.
2. Assign the Bragg reflexes to the respective lattice planes. Determine which Bravais lattice type it has.
3. Calculate the number of atoms in the unit cell.

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Theory (1/6)

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When X-rays of wavelength λ strike a mass of lattice planes of a crystal of spacing d at a glancing angle of θ, then the reflected rays will only be subject to constructive interference when Bragg's condition is fulfilled, i.e.:

$2\mathrm{d}\sin(\theta) = \mathrm{n}\lambda$ (n = 1, 2, 3, ...) (1)

Bragg's condition implies that all of the waves scattered at the atom are in phase and so amplify each other, whereas partial waves that are scattered in directions not fulfilling Bragg's conditions are of opposite phase and so extinguish each other. A more realistic way of looking at this must, however take the actual phase relationships of all of the partial waves scattered by the atom in a certain direction into consideration. When there are N atoms in a unit cell, then the total amplitude of the X-rays scattered by the cell is described by the structure factor F, which is calculated by summing up the atomic scattering factors f of the individual N atoms, taking their phases into account.

Theory (2/6)

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In general, the following is valid for F:

$\mathrm{F_{hkl}} = \sum_1^{\mathrm{N}} \mathrm{f_n} \cdot e^{2\pi i(\mathrm{hu_n}+\mathrm{kv_n}+\mathrm{lw_n})}$ (2)

where h, k, l = Miller indices of the reflecting lattice planes and $\mathrm{u_n}$, $\mathrm{v_n}$, $\mathrm{w_n}$ are the coordinates of the atoms in fractions of the particular edge lengths of the unit cell. As F is in general a complex number, the total scattered intensity is described by $|\mathrm{F_{hkl}}|^2$.

A cubic simple unit cell contains only one atom with the coordinates 000. According to equation (2), therefore, the structure factor F for this lattice type is given by:

$\mathrm{F} = \mathrm{f} \cdot e^{2\pi i(0)} = \mathrm{f};\ |\mathrm{F}|^2 = \mathrm{f}^2$ (3)

This means that F^2 is independent of h, k and l and all Bragg reflections can therefore occur.

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Theory (3/6)

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The unit cell of a cubic face-centered lattice has 4 atoms at 000, ½ ½ 0, ½ 0 ½ and 0 ½ ½. The unit cell of a cubic body-centered lattice has in comparison only 2 atoms at 000 and ½ ½ ½. When the lattice only consists of one sort of atom, then the following conditions are valid for the structure factor:

fcc Lattice

$|\mathrm{F}|^2 = 16\mathrm{f}^2$, with hkl only even or only odd; $|\mathrm{F}|^2 = 0$ with hkl mixed.

bcc Lattice

$|\mathrm{F}|^2 = 4\mathrm{f}^2$, with (h+k+l) even; $|\mathrm{F}|^2 = 0$ with (h+k+l) odd (4)

Theory (4/6)

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The situation is somewhat different when a lattice is made up of different sorts of atoms.

When, for example, an fcc lattice consists of the atoms A and B, whereby the A atoms lie at 000, ½ ½ 0, ½ 0 ½ and 0 ½ ½ , and the B atoms at ½ ½ ½ , 0 0 ½ , 0 ½ 0 and ½ 0 0, then the following additional condition is given for the structure factor F:

fcc Lattice with atoms A and B:

$|\mathrm{F}|^2 = 16(\mathrm{f_A} + \mathrm{f_B})^2$, with (h+k+l) even; $|\mathrm{F}|^2 = 16(\mathrm{f_A} - \mathrm{f_B})^2$ with (h+k+l) odd (5)

In such fcc lattices, when the atomic scattering factors f of the two atoms are almost equal ($\mathrm{f_A} \approx \mathrm{f_B}$), then 111 reflections will only be very weak, if they occur at all.

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Theory (5/6)

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For the cubic crystal system, the spacing d of the individual lattice planes with the indices (hkl) is obtained from the quadratic form:

$\frac{1}{d_{hkl}} = \frac{1}{a}(h^2 + k^2 + l^2)$ (a = lattice constant) (6)

From this and equation (1), with n = 1, the quadratic Bragg equation is obtained:

$\sin^2(\theta) = \frac{\lambda^2}{4a^2}(h^2 + k^2 + l^2)$ (7)

Theory (6/6)

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The following so-called strip-matching procedure can be used to index the individual reflexes of cubic crystals. Take logarithms in equation (7):

$\lg(a) = \lg\left(\frac{1}{2}\lambda\right) + \lg\left(\sqrt{h^2 + k^2 + l^2}\right) - \lg(\sin(\theta))$ (8)

Plot the experimentally determined values for lg $(\sin(\theta))$ on a strip of paper. In addition, plot the 2nd term on the right hand side of equation (8) on a separate strip of paper, taking all possible index triplets into consideration.

$\lg(a) = \lg\left(\frac{1}{2}\lambda\right) + \lg\left(\sqrt{h^2 + k^2 + l^2}\right)$ (9)

Now move the scales against each other until a position is found at which the graduations on the two strips match up to a great extent. The distance between the zero points of the two strips now gives the value of lg(a): Taking anti-logs gives the lattice constant a of the cubic system.

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Equipment

Position	Material	Item No.	Quantity
1	XR 4.0 expert unit, 35 kV	09057-99	1
2	XR 4.0 X-ray goniometer	09057-10	1
3	XR4 X-ray Plug-in Cu tube	09057-51	1
4	XR 4.0 X-ray structural analysis upgrade set	09145-88	1
5	Sodium chloride 250 g	30155-25	1
6	Vaseline 100 g	30238-10	1

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Setup and Procedure

Setup

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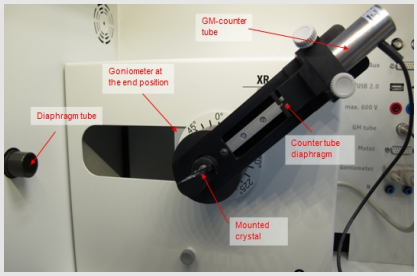
Connect the goniometer and the Geiger-Müller counter tube to their respective sockets in the experiment chamber (see the red markings in Fig. 1). The goniometer block with the analyser crystal should be located at the end position on the right-hand side. Fasten the Geiger-Müller counter tube with its holder to the back stop of the guide rails. Do not forget to install the diaphragm in front of the counter tube (see Fig. 2). Insert a diaphragm tube with a diameter of 2 mm into the beam outlet of the tube plug-in unit.

For calibration: Make sure, that the correct crystal is entered in the goniometer parameters. Then, select “Menu”, “Goniometer”, “Autocalibration”. The device now determines the optimal positions of the crystal and the goniometer to each other and then the positions of the peaks.

← Fig. 1: Connectors in the experiment chamber



↓ Fig. 2: Set-up of the goniometer

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Procedure (1/5)

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- Connect the X-ray unit via the USB cable to the USB port of your computer (the correct port of the X-ray unit is marked in Figure 3).
- Start the “measure” program. A virtual X-ray unit will be displayed on the screen.
- You can control the X-ray unit by clicking the various features on and under the virtual X-ray unit. Alternatively, you can also change the parameters at the real X-ray unit. The program will automatically adopt the settings.

Fig. 3: Connection of the computer

Procedure (2/5)

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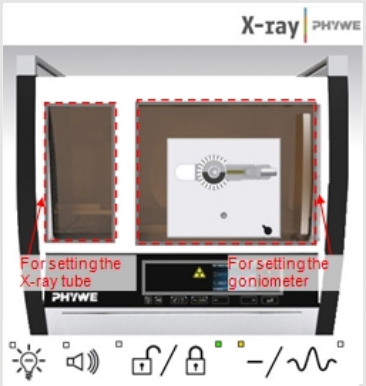

Fig. 4: Part of the user interface of the software

- Click the experiment chamber (see the red marking in Fig. 4) to change the parameters for the experiment.
- If you click the X-ray tube (see the red marking in Figure 4), you can change the voltage and current of the X-ray tube. Select the settings as shown in Figure 5.

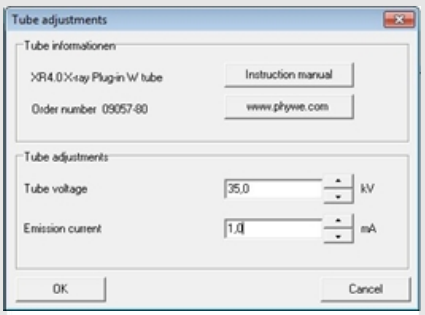

Fig 5: Voltage and current settings

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Procedure (3/5)

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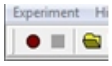
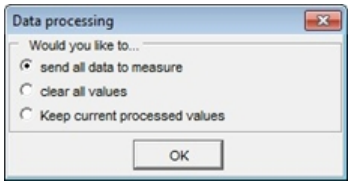
- Start the measurement by clicking the red circle: 
- After the measurement, the following window appears:



- Select the first item and confirm by clicking OK. The measured values will now be transferred directly to the “measure” software.
- At the end of this manual, you will find a brief introduction to the evaluation of the resulting spectra.

Procedure (4/5)

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Overview of the settings of the goniometer and X-ray unit:

- 1:2 coupling mode
- angle step width 0.1°
- Scanning range: 10° - 45°
- Anode voltage U_A = 35 kV UA=35kV; anode current I_A = 1 mA
- Scanning speed, when only the very intense reflex lines are to be recorded, then scanning can be relatively rapid at 10 s/°. For the identification of weaker lines, a scanning speed of at least 30 s/° is required for a better signal/noise ratio

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Procedure (5/5)

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Sample preparation:

The sample must be so finely powdered that no grains can be felt when a little of it is rubbed between finger and thumb. A relatively high sample concentration can be obtained by mixing the powder with a little vaseline. To do this, transfer a small amount of the sample onto a sheet of paper and use a spatula to knead it to a firm paste. To achieve the highest concentration of material as possible, use very little vaseline (a spatula tip of it). Fill the relatively solid sample paste into the specimen for powder samples and smooth it flush. Use the universal crystal holder to hold the specimen.

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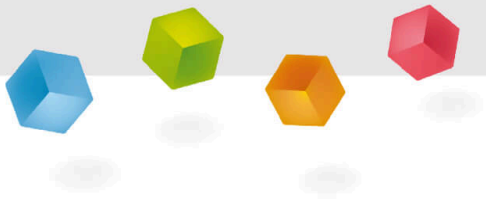

Evaluation

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Task 1

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Fig. 6 shows the Debye-Scherrer spectrum of sodium chloride (NaCl).

As no filter is used for the monochromatization of the X-rays, when individual lines are evaluated consideration must be given to the fact that the very intense lines that result from K_α-radiation are accompanied by secondary lines that result from the weaker K_β radiation. These pairs of lines can be identified by means of equation (1). It is namely approximately true with $\lambda(\mathrm{K}_\alpha) = 154.18\,\mathrm{pm}$ and $\lambda(\mathrm{K}_\beta) = 139.22\,\mathrm{pm}$:

$$\frac{\lambda(\mathrm{K}_\alpha)}{\lambda(\mathrm{K}_\beta)} = \frac{\sin(\theta_\alpha)}{\sin(\theta_{beta})} \approx 1.1$$

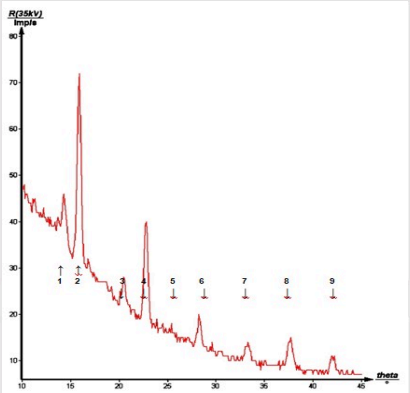

Fig. 6: Bragg $\mathrm{Cu} - \mathrm{K}_\alpha$ and $\mathrm{Cu} - \mathrm{K}_\beta$ lines of NaCl.

Task 1 (part 2)

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These values correspond to the quotients of the sinq values (Fig. 6) of the pairs of lines 2-1, 4-3 and 6-5, showing that the lines 1, 3, 5 and 7 originate from the CuK_β radiation.

The correctness of this conclusion can be shown by a control measurement (see Fig. 7) using the diaphragm tube with nickel foil to reduce the intensity of the K_β radiation. The reflexes in Fig. 6 that were previously assigned to the K_β lines are no longer to be seen. As the intensity of the K_β-radiation is also somewhat weakened by the Ni foil, the detection of reflexes of weak intensity at larger glancing angles is made difficult when this is used.

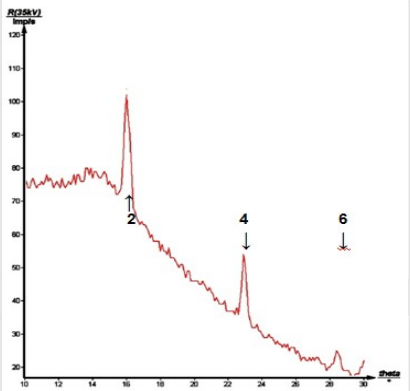

Fig. 7: Bragg-diagram of NaCl only with $\mathrm{Cu} - \mathrm{K}_\alpha$ beam (a nickel filter was used here)

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Task 2

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For reasons of clarity, it is assumed as a limitation in the following that NaCl does not form a simple unit cell, but rather an fcc or bcc lattice. Table 1 lists all of the possible index triplets possible in this case, i.e only non-mixed hkl combinations, or such for which (h + k + l) = 2n is true, were considered. The wavelength $\lambda(K_\alpha)$ = 154.18 pm was used to calculate values from equation (9).

hkl	$h^2+k^2+l^2$	$\lg(\sqrt{h^2+k^2+l^2})+\lg\left(\frac{1}{2}\lambda\right)$	hkl	$h^2+k^2+l^2$	$\lg(\sqrt{h^2+k^2+l^2})+\lg\left(\frac{1}{2}\lambda\right)$
111	3	2,126	213	14	2,460
200	4	2,188	004	16	2,489
112	6	2,276	114/033	18	2,515
012	8	2,339	313	19	2,526
013	10	2,387	024	20	2,538
113	11	2,408	323	22	2,558
222	12	2,427	224	24	2,577

Table 1: Permissible h,k,l index triplets for fcc and bcc lattices.

Task 2 (part 2)

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For the determination of the reflex angle from Fig. 6, zooming on the corresponding angle region and accurate determination of the main part of the line to two decimal places is recommended
Table 2 lists the glancing angles of the k_α- radiation reflexes determined from Fig. 6, as well as the corresponding sin values and lg(sin) values.

Line	θ [°]	$\sin(\theta)$	$\lg(\sin(\theta))$
2	15,890,2738		-0,5626
4	22,790,3874		-0,4119
6	28,300,4741		-0,3241
7	33,250,5483		-0,2610
8	37,700,6115		-0,2136
9	42,050,6698		-0,1741

Table 2: Glancing angles of the NaCl reflexes from Fig. 6.

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Task 2 (part 3)

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To obtain satisfactory accuracy from the graphical evaluation, the two scales should preferably be prepared as shown in Fig.8, equal but expanded.
The coincidence of the two scales shows that no mixed indexed triplets occur, but only even numbered hkl values.
Lattice constant a is determined from the logarithm of the difference in the zero points of the two scales. As Fig. 8 shows, the two sclaes coincide at the values 2.30 and -0.45, i.e. the difference is 2.75. Taking the anti-log of 2.75, a = 562.3 pm is obtained for the lattice constant (literature value; a = 563.0 pm). It can be shown that the unit must be “pm“ by an example, using equation (1) to calculate the lattice plane spacing d of any of the reflexes in Fig. 6.

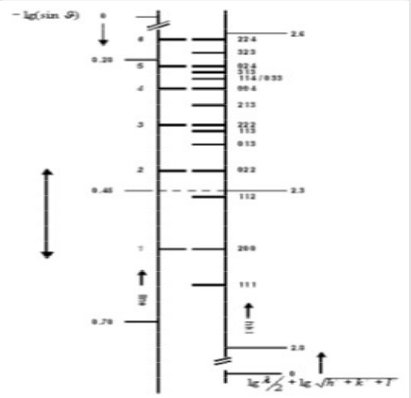

Fig. 8: Evaluation of the NaCl reflex lines using the stripmatching procedure

Task 3

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On dividing the total mass M of a unit cell by its volume V, the density ρ is given, so that:

$$\rho = \frac{M}{V} = n \cdot m \cdot \frac{1}{a^3} \text{ with } m = \frac{m_A}{N} \rightarrow n = \frac{\rho \cdot N \cdot a^3}{m_A} \qquad (11)$$

where n = the number of atoms or molecules in the unit cell; m = atomic/molecular mass; m_A = atomic/molecular weight;= $N = 6.022 \cdot 10^{23}$ = Avogadro’s number. The following are known values for NaCl, $\rho = 2.164\,g/cm^3$ and $m_A = 58.44\,g$. Using these values and a = 562.3 pm in equation (11), $n = 3.96 \approx 4$ is obtained, i.e. according to this, the unit NaCl cell contains not 2 but 4 atoms. This would mean that NaCl forms an fcc lattice. This contradiction to the above results can be clarified by returning to the considerations made with equation (4). Considering the fact that the atomic scattering factor f correlates linearly with the number of electrons of an atom, among others, and that Na (Z = 11) and Cl (Z = 17) have nearly the same scattering power, it follows from equation (5) that reflexes with odd numbered hkl triplets can only occur very weakly, if at all.The NaCl results of n = 4 and (h + k + l) = 2n can only be brought into agreement with an fcc lattice type.

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