Debye-Scherrer diffraction patterns of powder samples with a hexagonal lattice structure

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/5f75b94d3de4410003d84e8c

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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 polycrystalline powder sample of zinc 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 Debye-Scherrer pattern is automatically recorded. The evaluation of the pattern not only allows the Bragg reflexes to be assigned to the individual lattice planes and so also the corresponding Bravais lattice type to be obtained, but in addition results in values for their spacing as well as for the lattice constants of zinc and the number of atoms in the unit cell.

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 zinc powder sample as a function of the scattering angle.
2. Calculate the lattice constants of the substance from the angular positions of the individual Bragg lines.
3. Assign the Bragg reflexes to the respective planes of the zinc lattice and determine the Bravais lattice type of it.
4. Determine the number of atoms in the unit cell.

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Theory (1/3)

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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 (2/3)

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In general, the following is valid for F:

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

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 $|\mathrm{F_{hkl}}|^2$.

The unit cell of a hexagonal system with the most dense packing of spheres contains two atoms with positions (000) and $\left(\frac{2}{3}\,\frac{1}{3}\,\frac{1}{2}\right)$. According to equation (2), therefore, the structure factor F for this lattice type is given by:

$$\mathrm{F} = \mathrm{f}\left(e^{2\pi i(0)} + e^{2\pi i\left(\frac{2}{3}\mathrm{h}+\frac{1}{3}\mathrm{k}+\frac{1}{2}\mathrm{l}\right)}\right)$$

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Theory (3/3)

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Table 1 gives the selection rules for structure factor F.

h + 2k l		$\|F\|^2$
3n	odd	0
3n	even	$4f^2$
$3n \pm 1$	odd	$3f^2$
$3n \pm 1$	even	f^2

Table 1: Selection rules for the structure factor F of hexagonal crystal systems

n = 0, 1, 2, 3, 4, ...

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

$$\frac{1}{d_{hkl}} = \frac{4}{3}\left(\frac{h^2+hk+k^2}{a^2}\right) + \frac{l^2}{c^2}$$ (a, c = lattice constants) (4)

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

$$\sin^2(\theta) = \frac{\lambda^2}{4}\left(\frac{4}{3}\frac{h^2+hk+k^2}{a^2} + \frac{l^2}{c^2}\right)$$ (5)

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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	Vaseline 100 g	30238-10	1
6	Zinc,powder 500 g	31979-50	1

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Setup and Procedure

Setup

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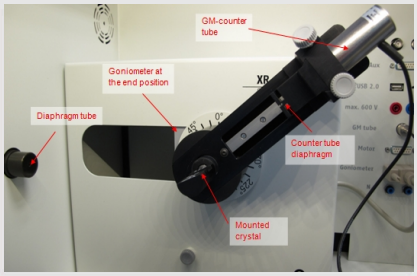
Connect the goniometer and the Geiger-Müller counter tube to their respective sockets in the experiment chamber (see the red markings in Fig. 1). The goniometer block with the analyser crystal should be located at the end position on the right-hand side. Fasten the Geiger-Müller counter tube with its 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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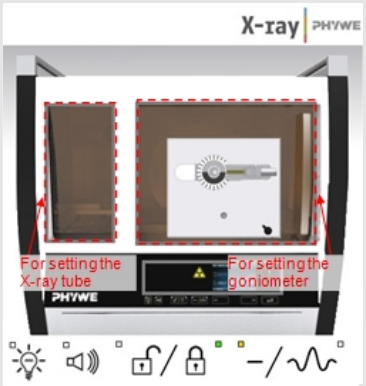

Fig. 4: Part of the user interface of the software

- 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.

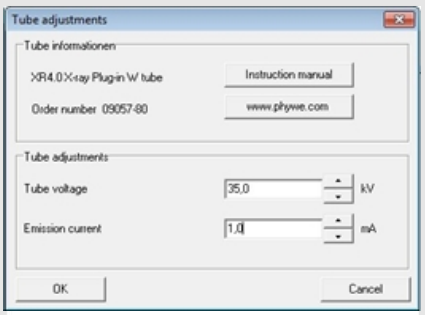

Fig 5: Voltage and current settings

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Procedure (3/5)

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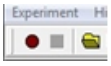
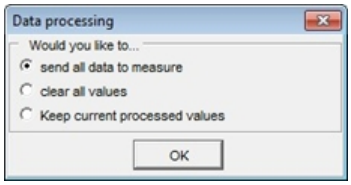
- Start the measurement by clicking the red circle: 
- After the measurement, the following window appears:



- 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 (3/5)

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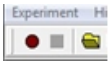
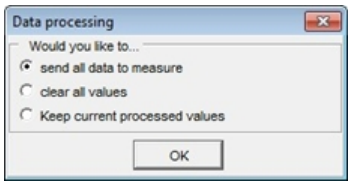
- Start the measurement by clicking the red circle: 
- After the measurement, the following window appears:



- Select the first item and confirm by clicking OK. The measured values will now be transferred directly to the “measure” software.
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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° - 60° bzw. Mit Filter: 10° - 28°
- 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 40 s/° is required for a better signal/noise ratio

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

Task 1

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Fig. 6 shows the Debye-Scherrer spectrum of germanium (Ge).

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 $\lambda(\mathrm{K}_\beta) = 139.22\,\mathrm{pm}$:

$$\frac{\lambda(\mathrm{K}_\alpha)}{\lambda(\mathrm{K}_\beta)} = \frac{\sin(\theta_\alpha)}{\sin(\theta_{beta})} \approx 1.1 \qquad (6)$$

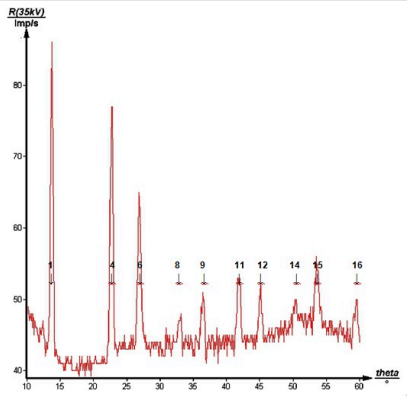

Fig. 6: Bragg $\mathrm{Cu} - \mathrm{K}_\alpha$ and $\mathrm{Cu} - \mathrm{K}_\beta$ lines of zinc.

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Task 1 (part 2)

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These values correspond to the quotients of the sinq values (Table 4) of the pairs of lines 2-1, 4-3, 6-5 and 8-7, which shows 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.

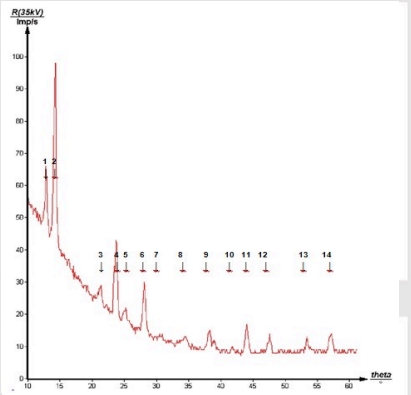

Fig. 7: Bragg-diagram of zinc only with $\mathrm{Cu-K}_\alpha$ beam (a nickel filter was used here)

Task 2 and 3

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For the evaluation of the spectrum, equation (5) is rearranged as follows:

$\sin^2(\theta) = \mathrm{A}(\mathrm{h}^2 + \mathrm{hk} + \mathrm{k}^2) + \mathrm{Bl}^2$ with $\mathrm{A} = \frac{\lambda^2}{4\mathrm{a}^2}$ and $\mathrm{B} = \frac{\lambda^2}{4\mathrm{c}^2}$ (7)

The value for A is determined solely by the hk lines. With l = 0, it follows from equation (7) that:

$\sin^2(\theta) = \mathrm{A}(\mathrm{h}^2 + \mathrm{hk} + \mathrm{k}^2)$ (8)

The permissible values for $(\mathrm{h}^2 + \mathrm{hk} + \mathrm{k}^2)$ are 1, 3, 4, 7, 9, 12, ... (see Table 2).

Divide the $\sin^2(\theta)$ values by 3, 4, 7, ... and search for quotients that are equal to each other, or to $\sin^2(\theta)$ values, as it can be assumed that these belong to the hk lines. Only the first reflex lines need be examined here, as these always belong to the low indexed lattice planes (see Table 3).

hk	10	11	12	02	13	02	23	1
$\mathrm{h}^2 + \mathrm{hk} + \mathrm{k}^2$	1	3	4	7	9	12	13	

Table 2: Permissible h,k combinations

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Task 2 and 3 (part 2)

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The first lattice constant a:

- It is clear from Table 3 that for lines 3 and 8 the bold face values are nearly in agreement.
- On calculating the mean value of these, A = 0.1117 is obtained
- Using this value for A and $\lambda(\mathrm{K}_\alpha) = 154.18\,\mathrm{pm}$, it follows from equation (6) that for the first lattice constant: a = 266.3 pm
- On carrying out an experiment that suggests itself, i.e. by assigning the value A = 0.1117 to line 3 of index 100, then the $\sin^2(\theta)$ value of line 8 must be assigned to the 110 reflex, as this is about 3 times the corresponding value of line 3.
- Now subtract A, 3A, 4A etc. from the $\sin^2(\theta)$ values and search for Bl^2 values that are in a ratio to each other of 1, 4, 9, 16 etc.:

Task 2 and 3 (part 3)

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Line	θ [°]	$\sin(\theta)$	$\sin^2(\theta)$	$\sin^2(\theta)/3$	$\sin^2(\theta)/4$	$\sin^2(\theta)/7$	hkl
1	16,25	0,2798	0,0783	0,0261	0,0196	0,0112	
2	18,18	0,3120	0,0973	0,0324	0,0243	0,0139	
3	19,58	0,3351	0,1123	0,0374	0,0281	0,0160	1 0 0
4	21,71	0,3699	0,1368	0,0456	0,0342	0,0195	
5	24,17	0,4094	0,1676	0,0559	0,0419	0,0239	
6	27,28	0,4583	0,2101	0,0700	0,0525	0,0300	
7	31,38	0,5207	0,2711	0,0904	0,0678	0,0387	
8	35,26	0,5773	0,3333	0,1111	0,0833	0,0476	1 1 0
9	38,39	0,6210	0,3857	0,1286	0,0964	0,0551	

Table 3:Evaluation of the K_α- lines for the determination of lattice constant a.

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Task 2 and 3 (part 4)

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The second lattice constant c:
We find from Table 4 that the bold face values 0.0251, {1/4(0.0973+0.0984+0.0976+0.0954) = 0.0972}, 0.2216 and 0.3857 approximately fulfill this. B can so be determined from the relationships: 0.0251 = 1^2B, 0.0972 = 2^2B, 0.2216 = 3^2B, 0.3857 = 4^2B. A mean value of B = 0.0245 is found.

The second lattice constant for the hexagonal lattice can be obtained using this value for B and equation (6): c = 492.5 pm. In addition, it follows that the lines 2 and 9 have the indices 002 and 004, because with $(h^2 + hk + k^2)$ = 0, from equation (6):
Line 2: $\sin^2(\theta)$ = 0.0972 = $B \cdot l^2$ = 0.0245·l^2 $\rightarrow$ l = 1.99 $\approx$ 2
Line 9: $\sin^2(\theta)$ = 0.3857 = $B \cdot l^2$ = 0.0245·l^2 $\rightarrow$ l = 3.96 $\approx$ 4

((hkl) can only be integer numbers)
Line 4, for example, has the indices h = 1, k = 0 and l = 1, as: $\sin^2(\theta)$ = 0.1368 $\approx$ A + B = 0.1362 or $\sin^2(\theta)$ - A = 0.0251 $\approx$ B = 0.0245.

Task 2 and 3 (part 5)

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The indices of all other lines with the exception of lines 1, 5 and 7 can be correspondingly found, as given in Table 4.

Line	$\sin^2(\theta)$	$\sin^2(\theta) - A$	hkl
1	0,0783		002 (K_β)
2	0,0973		002
3	0,1123	0,0006	100 and 101 (K_β)
4	0,1368	0,0251	101
5	0,1676	0,0559	102 (K_β)
6	0,2101	0,0984	102
7	0,2711	0,1594	110 (K_β)

Line	$\sin^2(\theta)$	$\sin^2(\theta) - A$	$\sin^2(\theta) - 3A$	$\sin^2(\theta) - 4A$	hkl
8	0,3333	0,2216			110
9	0,3857	0,2740	0,0506		004
10	0,4327	0,3210	0,0976		112
11	0,4712	0,3595	0,1361	0,0244	201
12	0,5422	0,4305	0,2071	0,0954	202
13	0,6643	0,5526	0,3292	0,2175	203
14	0,7223	0,6106	0,3872	0,2755	105

Table 4:Evaluation of the reflex lines for the determination of lattice constant c and the assignment of Miller indices.

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