Determination of the lattice constants of a monocrystal

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/5f9312aef8aae70003e978cb

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General information

Application

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 for this experiment is found in the Theory section.

Main principle

Polychromatic X-rays impinge on a monocrystal under various glancing angles. The rays are reflected by the lattice planes of the monocrystal. An energy detector is only used to measure those radiation parts that interfere constructively. The lattice constant of the crystal is determined with the aid of the various orders of diffraction and the energy of the reflected rays.

Other information (2/2)

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Learning objective

The goal of this experiment is to get to determine the gitter constant of a monocrystal.

Tasks

1. Determine the energy of the X-rays that are re-flected at the lattice planes of the LiF-crystal for various glancing angles or diffraction orders.
2. Calculate the lattice constant of the LiF-crystal based on the glancing angles and associated energy values.

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Theory (1/3)

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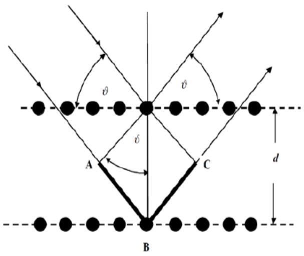

Fig. 1: Constructive interference during the reflexion on lattice planes of crystals

The X-rays that hit the monocrystal under the glancing angle θ (see Fig. 1) can only be observed in a reflection if the incident and reflected rays of the same wavelength interfere constructively. This means, however, that the path difference $\Delta$of the rays must be an integer multiple of the wavelength (in Fig. 2, Δ is represented by the segments that are marked with thick lines):

$$\Delta = 2\mathrm{d}\sin(\theta) = \mathrm{n}\lambda \qquad (1)$$

The condition in (1) is known as the Bragg condition. (d distance between the lattice planes, θ glancing angle, λ wavelength, and n = 1, 2, 3, ...). If monoenergetic rays with a wavelength λ hit the family of lattice planes of the monocrystal and if the glancing angle is large (larger path difference Δ), a 2nd order diffraction can be observed in accordance with (1) with n = 2.

Theory (2/3)

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If, however, polychromatic rays hit the crystal and if the glancing angle is fixed (fixed path difference Δ), the reflected ray will have parts with the wavelengths $\lambda, \frac{1}{2}\lambda, \frac{1}{3}\lambda, \ldots$ in accordance with (1), since in this case the path difference corresponds to $\lambda, 2\frac{1}{2}\lambda, 3\frac{1}{3}\lambda, \ldots$(see Fig. 2). This situation can only be proved with an energy detector, but not with a Geiger-Müller counter tube that is usually operated in the triggering mode. If one replaces the wavelength in (1) by the associated energy $\mathrm{E} = \mathrm{h}\nu$ and $\mathrm{c} = \lambda\nu$, lead to:

$$\mathrm{E_n} = \mathrm{n}\frac{\mathrm{hc}}{2\mathrm{d}\sin(\theta)} \qquad (2)$$

With a fixed glancing angle θ equation (2) leads to:

$$\mathrm{E_n} = \mathrm{k}\cdot\mathrm{n} \text{ and } \frac{\mathrm{E_n}}{\mathrm{E_1}} \qquad (3)$$

Planck's quantum of action	h = $6.626 \cdot 10^{-34}$ Js
Speed of light in vacuum	$\mathrm{c} = 2.998 \cdot 10^{8}\ \mathrm{m/s}$
Photon frequency	ν

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Theory (3/3)

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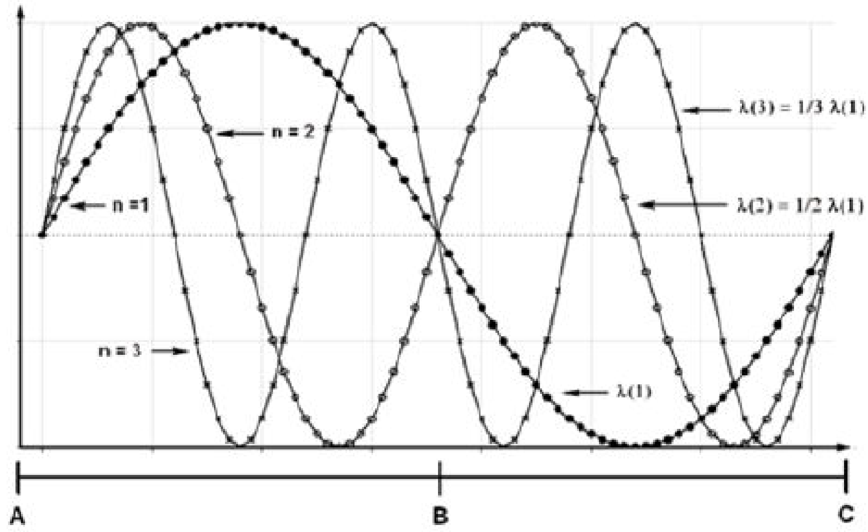

Fig. 2: Expanded path difference $\Delta = \mathrm{ABC}$ to show various diffraction orders with $\mathrm{n} = 1(\lambda), \mathrm{n} = 2(2\frac{1}{2}\lambda)$ and $\mathrm{n} = 3(3\frac{1}{3}\lambda)$

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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 material upgrade set	09165-88	1

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Setup and Procedure

Setup (1/2)

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- Screw the adapter ring onto the inlet tube of the energy detector and connect the signal and supply cables to the corresponding ports of the detector with the aid of the right-angle plugs.
- Connect the signal and supply cables to the corresponding ports in the experiment chamber of the X-ray unit. In Figure 3, the port for the signal cable is marked in red and the port for the supply cable is marked in green. Connect the external X RED ports of the x-ray unit (see Fig. 4) to the multi-channel analyser (MCA). Connect the signal cable to the "Input" port and the supply cable to the "X-Ray Energy Det." port of the MCA.

Fig. 3: Connectors in the experiment chamber

Fig. 4: Connection of the multi-channel analyser

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Setup (2/2)

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- Secure the energy detector in the holder of the swivel arm of the goniometer. Lay the two cables with sufficient length so that the goniometer can be swivelled freely over the entire range.
- Connect the multi-channel analyser and computer with the aid of the USB cable.
- Insert the tube with the 2-mm-aperture.
- Bring the goniometer block and the detector to their respective end positions on the left. Bring the detector to the 90° position in the 2:1 coupling mode (Fig. 5).

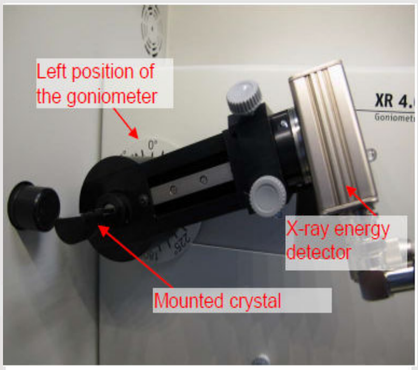

Fig. 5: Goniometer set-up

Procedure (1/4)

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- Bring the goniometer block and the detector to their respective end positions on the right.
- Insert the tube with the 1mm-aperture into the exit tube of the X-ray tube.
- With the X-ray unit switched on and the door locked, bring the detector to the 0° position. Then, shift the detector by some tenths degree out of the zero position in order to reduce the total rate.
- Operating data of the tungsten X-ray tube: Select an anode voltage U_A = 25 kV and an anode current I_A = 0.02 mA and confirm these values by pressing the “Enter” button.
- Switch on the X-radiation

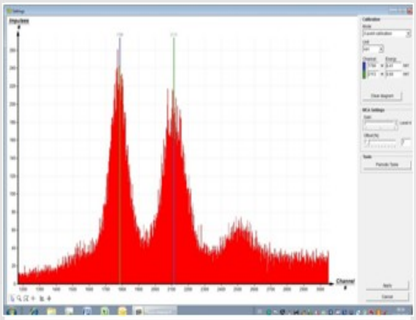

Fig. 6: calibration of the multi-channel analyser

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Procedure (2/4)

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- In the MEASURE program, select “Multi channel analyser” under “Gauge”. Then, select “Settings and calibration”. After the “Calibrate” button has been clicked, a spectrum can be measured. The counting rate should be < 300 c/s. Energy calibration settings: - 2-point calibration, - Unit = keV, Gain = 2 – Set the offset so that low-energy noise signals will be suppressed (usually a few per cent are sufficient), See Fig 6.
- Measuring time: 5 minutes. Use the timer of the X-ray unit.
- Make the two coloured calibration lines congruent with the line centres of the two characteristic X-ray lines. The corresponding energy values (see e.g. P2544705) $\mathrm{E(L_3M_5/L_3M_4)}$ = 8,41keV and $\mathrm{E(L_2N_4)}$ = 9,69 keV are entered into the corresponding fields, depending on the colour. (Note: Since a separation of the lines $\mathrm{L_3M_4}$ and $\mathrm{L_3M_5}$ Lines is not possible, the mean value of both lines is entered as the energy of the line).
- Name and save the calibration.

Procedure (3/4)

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Measurement of spectra with different orders of diffraction

- The goniometer block with the inserted LiF-crystal is at the left stop.
- The detector is at the front on the guide rods.
- Use the 1mm-diameter tube.
- Operate the crystal and the detector in the 2:1 mode.
- Use the parameters that were also used for the energy calibration.
- Record spectra at glancing angles from 10° - 42° in steps of 2°.
- Adjust the anode voltage $\mathrm{U_A} = 35\,\mathrm{kV}$ and athe anode current $\mathrm{I_A}$ so that the counting rates of the various spectra are $\approx 400\,\mathrm{c/s}$.

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Procedure (4/4)

Evaluation of the measurement curves

- In order to determine the line energy, switch from the bar display to the curve display. To do so, click “Display options” and then “Interpolation and straight lines”.
- Extend the relevant line section with the aid of the zoom function
- Then, select the curve section with Open the window “Function fitting Then, select “Scaled normal distribution” and confirm.
- Find the line centroid of the normal distribution with “Peak analysis” or determine it with the function “Survey”

Evaluation

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Task 1

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Figure 7 shows the Bragg reflexes for various glancing angles. If the glancing is increased (increase of Δ), more Bragg reflexes can be measured due to the constructive interference. If Δincreases, their energy decreases, since the associated wavelengths become increasingly larger. The intensity in the low-energy range of the primary ray is very low. In addition, the spectral parts of larger wavelengths are absorbed to a higher extent by the glass bulb of the X-ray tube. Both of these become noticeable in the decrease of intensity of the reflex lines up to their disappearance (see the line with n = 1 in Fig. 7d and 7c). The weak line at E = 8 keV must be assigned to the $\mathrm{Cu-K_\alpha}$ fluorescence radiation. When the monoenergetic photons, which are reflected by the crystal, penetrate the detector housing, they may cause fluorescence radiation around the materials that are used in the detector assembly. This radiation is also measured by the detector. Table 1 shows the evaluation of the spectra. Reflex lines with n = 5 are ignored due to their low intensity.Columns B - E show the energy values $\mathrm{E_n}$ of the various diffraction orders with different glancing angles (column A) that were determined during the experiment. Columns F - I show the corresponding values $\mathrm{E_n/n}$, whose rounded mean values are given in column J.

Task 1 (part 2)

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A	B	C	D	E	F	G	H	I	J	K
ϑ/°	n = 1 E_1 / keV	n = 2 E_2 / keV	n = 3 E_3 / keV	n = 4 E_4 / keV	$\frac{E_1}{1}$ /keV	$\frac{E_2}{2}$ /keV	$\frac{E_3}{3}$ /keV	$\frac{E_4}{4}$ /keV	E_{mittel}/keV	d/pm
10	17,69				17,69				17,69	201,8
12	14,80	29,60			14,80	14,80			14,80	201,5
14	12,70	25,43			12,70	12,72			12,71	201,6
16	11,17	22,40			11,17	11,20			11,19	201,0
18	9,93	20,00			9,93	10,00			9,97	201,2
20	8,94	18,05			8,94	9,03			8,98	201,8
22	8,14	16,52	24,87		8,14	8,26	8,29		8,23	201,1
24	7,53	15,20	22,88		7,53	7,60	7,63		7,59	200,8
26	6,94	14,05	21,17	28,19	6,94	7,03	7,06	7,05	7,02	201,5
28	6,51	13,16	19,82	26,54	6,51	6,58	6,61	6,64	6,59	200,4
30	6,11	12,36	18,62	24,89	6,11	6,18	6,21	6,22	6,18	200,6
34	5,43	11,04	16,63	22,22	5,43	5,52	5,54	5,56	5,51	201,2
38	4,96	10,00	15,10	20,31	4,96	5,00	5,03	5,01	5,00	201,4
42	-	9,2	13,84	18,56	-	4,60	4,61	4,64	4,62	200,5

Table 1: Determination of the interplanar spacing $\mathrm{d_{200}}$ of LiF

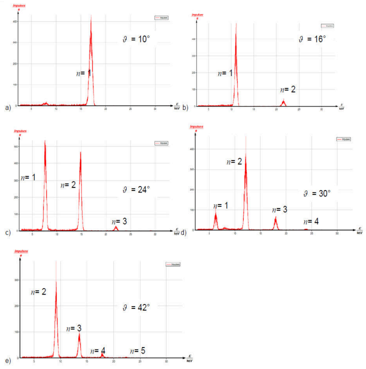

Fig. 7: Bragg reflexes with an increasing order of diffraction and various glancing angles.

a: θ = 10°, b: θ = 16°, c: θ = 24°, d: θ = 30°, e: θ = 42°

In Fig. e, the reflex with n = 1 cannot be observed.

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Task 2

Column K shows the value for the interplanar spacing d that was calculated with the aid of (2).

For this (100)-oriented LiF-monocrystal, the following rounded value results:

$\mathrm{d_{200}(LiF) = (201.2 \pm 0.5)\,pm}$; $\Delta \mathrm{d/d} \approx 0.3\,\%$; (literature value $\mathrm{d_{200} = 201.4\,pm}$).

Accordingly, the edge length of the cubic LiF-lattice $\mathrm{d_{200} = 402.8\,pm}$

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