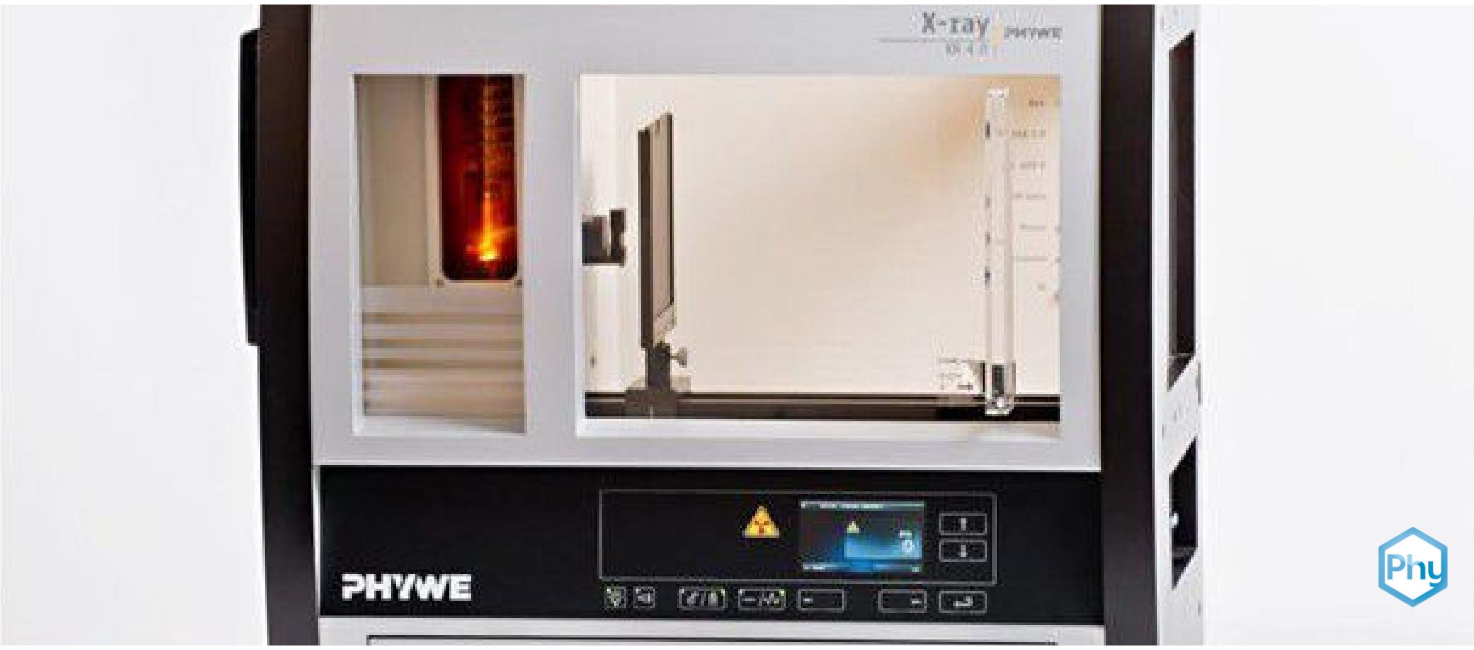
X-ray investigation of crystal structures / Laue method



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/5f608ee77e9d5b0003e1e805

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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 for this experiment is found in the Theory section.

Main principle

Laue diagrams are produced when monocrystals are irradiated with polychromatic X-rays. This method is primarily used for the determination of crystal symmetries and the orientation of crystals. When a LiF monocrystal is irradiated with polychromatic X-rays, a characteristic diffraction pattern results. This pattern is photographed and then evaluated.

Other information (2/2)

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Learning objective

The goal of this experiment is to investigate the Laue pattern of a LiF monocrystal.

Tasks

1. Take a photograph of the Laue pattern of a LiF monocrystal.
2. Assign the Laue reflections to the lattice planes of the crystal.

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Theory (1/3)

Laue diagrams are produced when monocrystals are irradiated with polychromatic X-rays. This method is used mainly for the determination of crystal symmetries and the orientation of crystals. A complete analysis of the diagrams is only possible with simple crystal structures.
A necessary, although insufficient, condition for the constructive reflection at the various lattice planes is the Bragg condition:

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

(d = interplanar spacing; θ = glancing angle; λ = wavelength; n = 1, 2, 3, ...)

With the lattice constant a of a cubic crystal, the following is valid for the spacing d(hkl) between the individual lattice planes:

$$\mathrm{d(hkl)} = \frac{\mathrm{a}}{\sqrt{\mathrm{h}^2+\mathrm{k}^2+\mathrm{l}^2}} \qquad (2)$$

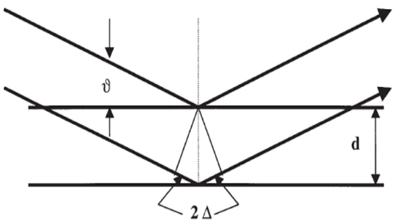

Fig. 1: Bragg scattering on a pair of lattice planes

Theory (2/3)

If L is the distance between a reflection and the centre of the Laue pattern, and D the distance between the film and the sample (Fig. 2), then the glancing angle θ_{exp} that is determined in an experimental manner is:

$$\theta_{\mathrm{exp}} = \frac{1}{2}\arctan\left(\frac{\mathrm{L}}{\mathrm{D}}\right);\ \mathrm{L} = \sqrt{\mathrm{y}^2+\mathrm{z}^2} \qquad (3)$$

y and z are the distances of the reflection in a system of rectangular coordinates with its origin in the centre of the pattern.

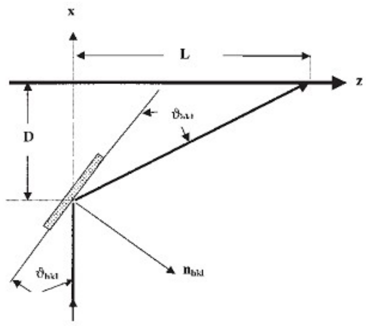

Fig. 2: Scattering geometry of a Laue pattern. The y-axis is in the plane of the film and is perpendicular to the x,z plane.

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Theory (3/3)

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If the X-ray beam coincides with a certain crystallographic direction [h*,k*,l*] (here, the [100] direction) and if it impinges on a crystal plane (h,k,l), then the angle of incidence α (see Fig. 3) is determined by the scalar product of the normal vector of the plane and the incident vector.

$$\cos(\alpha) = \frac{\mathrm{hh}^* + \mathrm{kk}^* + \mathrm{ll}^*}{\sqrt{(\mathrm{h}^2+\mathrm{k}^2+\mathrm{l}^2)\cdot((\mathrm{h}^*)^2+(\mathrm{k}^*)^2+(\mathrm{l}^*)^2)}}$$

Then, the following is valid for the glancing angle: $\theta_{\mathrm{cal}} = 90^\circ - \alpha$

According to the addition theorem and with (h*,k*,l*) = (100), it follows from (4) that:

$$\sin(\theta) = \frac{\mathrm{h}}{\sqrt{\mathrm{h}^2+\mathrm{k}^2+\mathrm{l}^2}} \qquad (5)$$

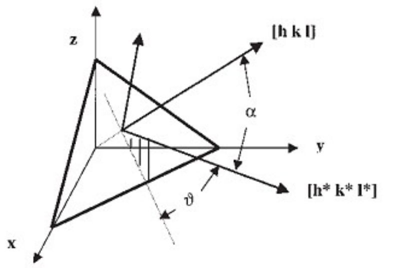

Fig. 3: Reflection on a lattice plane with random orientation.

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Equipment

Position	Material	Item No.	Quantity
1	XR 4.0 expert unit, 35 kV	09057-99	1
2	XR4 X-ray Plug-in Cu tube	09057-51	1
3	XR 4.0 X-ray structural analysis upgrade set	09145-88	1

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Setup and Procedure

Procedure (1/3)

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Prior to starting the experiment, take the goniometer out of the experiment chamber.
Then, insert the diaphragm tube with a diameter of 1 mm into the beam outlet of the X-ray plug-in unit. Add the crystal holder for Laue patterns. Install the LiF crystal with its two pins in the holder so that the rounded sides of the crystal holder face the X-ray tube. Position the film in darkness in the film holder (see fig. 4) and confirm that the holder is firmly closed. Fix the holder into the holder of the fluorescent screen and position it on the internal optical bench at a distance D = 1.5-2 cm from the crystal. The precise determination of this distance is very important for the subsequent evaluation. The film plane should be parallel to the crystal surface.
The X-ray tube is used at maximum power (anode voltage U_A = 35 kV, anode current I_A = 1 mA). The exposure time of atleast 30 min (2 hours for weaker reflections) can be set and activated as follows:

Fig. 4: Position of the film in the film holder

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Procedure (2/3)

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Fig. 5

Fig. 6

- Select the tube operating parameters under “X-ray parameters” and confirm them with “Enter”.
- Under “Menu”, select “Timer” (Fig. 5) → "Duration”. Set the desired time with the aid of the arrow buttons. Confirm with “Enter”.
- The window “Mode” appears. Select “On” and confirm with “Enter” (Fig. 6).
- To start the experiment, close and lock the sliding door and press the button under “Start” (Fig. 7).

Fig 7

Procedure (3/3)

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The irradiation starts. It will stop automatically after the preset exposure time. On the display, the remaining time can be observed based on a backwards running clock and a display bar.
X-ray films must be developed in a darkroom, following the instructions on the packaging. Then, the films are rinsed in a water bath before they are fixed for approximately 10 minutes. After that, the films are re-watered for 10 minutes and then dried in the air. Please refer to the instructions of use of the X-ray film for details concerning their use.

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Evaluation

Task 1

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Take a photograph of the Laue pattern of a LiF monocrystal.

Figure 8 shows the Laue diagram of a LiF(100) monocrystal with a face-centre cubic crystal lattice (fcc). If the diffraction pattern is rotated by 90° around the direction of the primary beam, it is again brought to coincidence. Since the primary beam impinges perpendicularly on the (100)-plane of the LiF crystal, the crystal direction [100] is a fourfold axis of symmetry. The intensity of the reflections depends on the reflecting crystal surface as well as on the spectral intensity distribution of the X-rays.

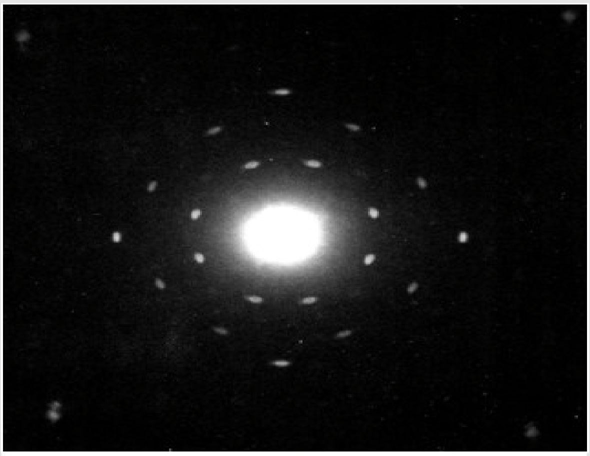

Fig. 8: Laue pattern of the LiF (100) crystal.

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Task 2

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Assign the Laue reflections to the lattice planes of the crystal.

The glancing angle θ_{cal} is calculated from (5) for all of the planes with low (h,k,l) indices. The angle θ_{exp} is determined using (3) based on the diagram. The assignment of the reflections to the lattice planes is found when the angles coincide and when the condition k/l = y/z is fulfilled, with z and y being the coordinates of the reflections.
A final control can be performed as follows. In accordance with the Duane-Hunt law of displacement (see experiment P2540905), the beginning of the bremsspectrum is given by the minimum wavelength $\lambda_{\mathrm{min}} = 1.24 \cdot 10^{-6} / \mathrm{u_A}\ [\mathrm{m}]$. For an accelerating voltage $\mathrm{u_A}$ = 35 kV, the following is true: λ_{min} = 35.5 pm. This means that for the assignment of the reflections to the lattice planes, only X-rays with a wavelength of \mathrm{\lambda} > 35.5 pm can play a role.

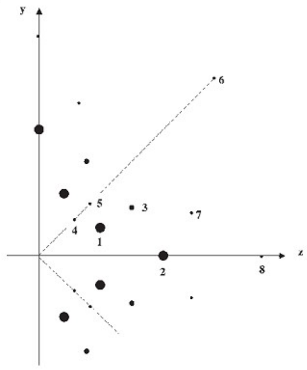

Fig. 9: Schematic representation of the Laue reflections.

Task 2 (part 2)

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Figure 9 shows the location of the reflections in a different manner. For reasons of symmetry, the evaluation can be restricted to 1/8 of the diagram. The other reflections are obtained by permutation of the indices and a change of the sign. Reflection no. 4 and 8 are only very slightly visible in the original photograph. For them a longer exposure time is necessary. Table 1 shows the result of the evaluation. It becomes clear that the reflections are visible only if the Miller indices are either all odd or all even. This is a characteristic feature of a face-centred cubic crystal lattice.

Reflection no.	y [mm]	z [mm]	L [mm]	θ_{exp} [°]	hkl	θ_{cal} [°]	k/l	y/z	d [pm]	λ [pm]
1	4.0	12.5	13.75	17.29	113	17.55	0.33	0.32	121.4	72.2
2	0.0	25.5	25.5	26.66	204	26.57	0	0	100.7	90.4
3	9.75	19.0	21.25	24.17	224	24.09	0.5	0.51	82.2	67.3
4	6.75	6.75	9.5	13.34	133	13.26	1	1	92.4	42.6
5	10.75	10.75	15.5	19.33	244	19.47	1	1	90.1	59.6
6	38.75	38.75	54.5	53.30	111	35.26	1	1	232.6	268.8
7	7.0	34.0	35.5	30.75	315	30.47	0.2	0.2	68.1	69.6
8	0.0	45.75	45.75	33.72	406	33.69	0	0	55.8	62.0

Table 1: Evaluation of the Laue diagram

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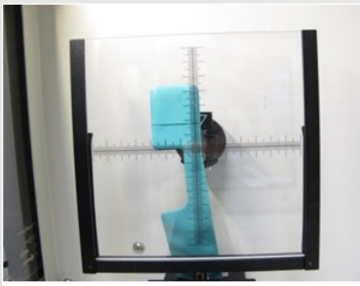
Appendix

Alternative Procedure (1/2)



Set-up in the X-ray unit

Data

Cu X-ray plug-in unit	09057-50
Tube voltage:	35 kV
Beam current:	1 mA
Diaphragm:	1 mm
Exposure time:	10-30 minutes

The position of the screen is determined with the aid of the mm scale on the optical bench.

Taking a Laue photograph with the aid of self-developing X-ray film

A monocrystal X-ray structure analysis can be performed live during a lecture with the aid of self-developing X-ray films in combination with the XR 4.0 expert unit. If a Cu X-ray tube is used, the photography only takes 12.5 minutes. The development itself takes only 2 to 3 min.

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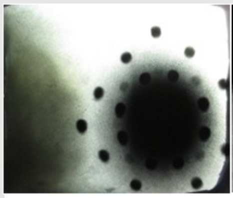
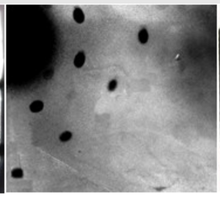
Alternative Procedure (2/2)

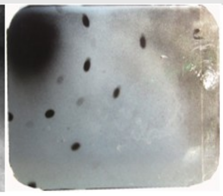
The X-ray film is not positioned centrally in front of the crystal. Instead, it is offset, since only a quadrant of the diagram is sufficient for the evaluation. The picture should be enlarged in order to evaluate it. We recommend scanning the photo and then enlarging it digitally.
As far as the development of the film is concerned, please refer to the instructions for use that are enclosed with the films. We recommend developing the film for 2 minutes instead of only 50 seconds. It is very important to hold the developed film under flowing water once it has been taken out of the wrap. Do not dry it with towels. Only let it air-dry.



Exposure time: 30 minutes Screen at 4.7 cm



Exposure time: 20 minutes Screen at 4.7 cm



Exposure time: 12.5 minutes Screen at 5.5 cm

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