Related topics

Characteristic X-radiation, Bravais lattices, reciprocal lattices, Miller indices, atomic form factor, structure factor, and Bragg scattering

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.

Equipment

1	XR 4.0 expert unit 35 kV	09057-99
1	X-ray Plug-in module with W X-ray tube	09057-80
1	X-ray Lithium fluoride monocrystal, mounted	09056-05
1	X-ray Crystal holder for Laue diffraction	09058-11
1	X-ray Diaphragm tube d = 1mm	09057-01
1	Vernier caliper, plastic	03014-00
1	XR 4.0 X-ray film, 100 × 100 mm²	09058-23
1	XR 4.0 X-ray Bag for x-ray films, 10 pieces	09058-22
1	X-ray-film developer for 4.5 l	06696-20
1	X-ray-film fixing for 4.5 l	06696-30
3	Laboratory tray, PP, 18 x 24 cm	47481-00
1	X-ray optical bench	09057-18
1	X-ray film holder	09057-08
1	Slide mount for optical bench, h = 30 mm	08286-01

This experiment is included in the upgrade set “XRS 4.0 X-ray structural analysis”.
Note: This experiment can also be performed with a copper or molybdenum X-ray tube. Instead of the X-ray films 09058-23, self-developing x-ray films (9057-20) can be used for the experiment. For more details, see appendix

Fig. 1: P2541601

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.

Procedure

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. 2) 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). In the case of the tungsten tube, the exposure time is 15-30 minutes. It can be set and activated as follows:

- Select the tube operating parameters and confirm them with “Enter”.
- Under “Menu”, select “Timer” (Fig. 3) → “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. 4).
- To start the experiment, close and lock the sliding door and press the button under “Start” (Fig. 5).

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.

Fig. 2: Position of the film in the film holder

Fig. 3

Fig. 4

Fig. 5

If you use a molybdenum or copper X-ray tube:

- Select an exposure time of at least 30 minutes.
- In order to be able to see weaker reflections, select an exposure time of up to 120 minutes.

Theory

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:

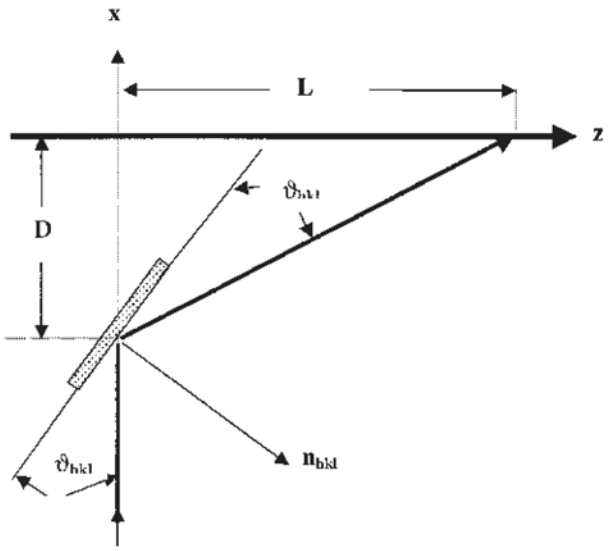


Fig. 6: Scattering geometry of a Laue pattern. The y-axis is in the plane of the film and is perpendicular to the x,z plane.

$$2d \sin \vartheta = n\lambda \text{ ; } (n = 1, 2, 3,\ldots) \quad (1)$$

(d = interplanar spacing, ϑ = glancing angle, λ = wavelength, and 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:

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

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. 6), then the glancing angle ϑ_{exp} that is determined in an experimental manner is:

$$\vartheta_{\text{exp}} = \frac{1}{2}\arctan\frac{L}{D}; \quad L = \sqrt{y^2 + z^2} \quad (3)$$

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

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. 7) is determined by the scalar product of the normal vector of the plane and the incident vector.

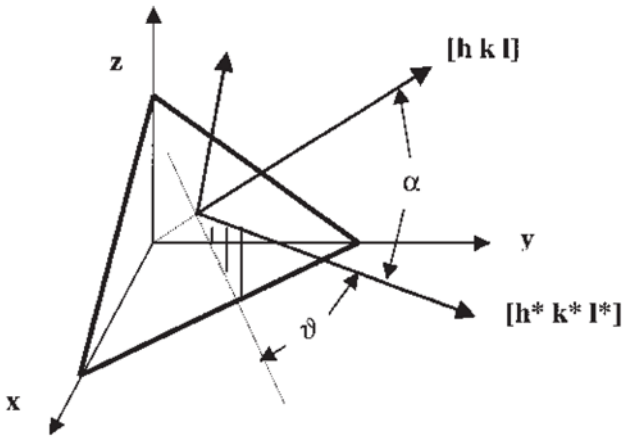


Fig. 7: Reflection on a lattice plane with random orientation.

$$\cos\alpha = \frac{h\,h^* + k\,k^* + l\,l^*}{\sqrt{\left(h^2 + k^2 + l^2\right)\cdot\left((h^*)^2 + (k^*)^2 + (l^*)^2\right)}}$$

Then, the following is valid for the glancing angle: $\vartheta_{cal} = 90° - \alpha$.

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

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

Evaluation

Task 1: 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.

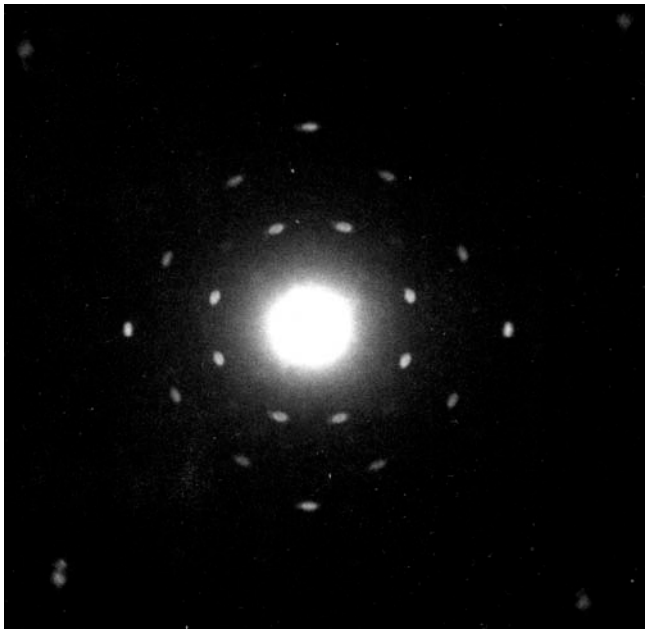


Fig. 8: Laue pattern of the LiF (100) crystal. Operating values of the tungsten X-ray tube: Accelerating voltage U_A = 35 kV; anode current I_A = 1 mA; distance between the crystal and film D = 15 mm; exposure time: t = 15 min

Task 2: 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 ($\vartheta_{cal} = \vartheta_{exp}$) 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 P2540901), the beginning of the bremsspectrum is given by the minimum wavelength $\lambda_{min} = 1.24\cdot10^{-6}/U_A$ [m]. For an accelerating voltage 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 λ > 35.5 pm can play a role.

Fig. 9: Schematic representation of the Laue reflections.

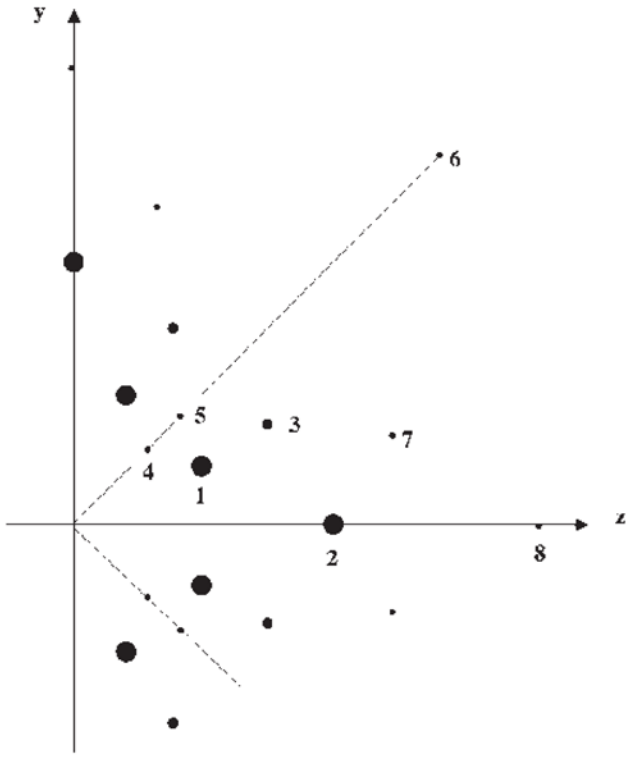


Figure 9 shows the location of the reflections in a different manner. For reasons of symmetry, the evaluation can be restricted to 1/8 of the diagram. The other reflections are obtained by permutation of the indices and a change of the sign. Reflection nos. 4 and 8 are only very slightly visible in the original photograph. They can only be seen clearly when a longer exposure time is used.

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 (see experiment 2541301).

Table 1: Evaluation of the Laue diagram

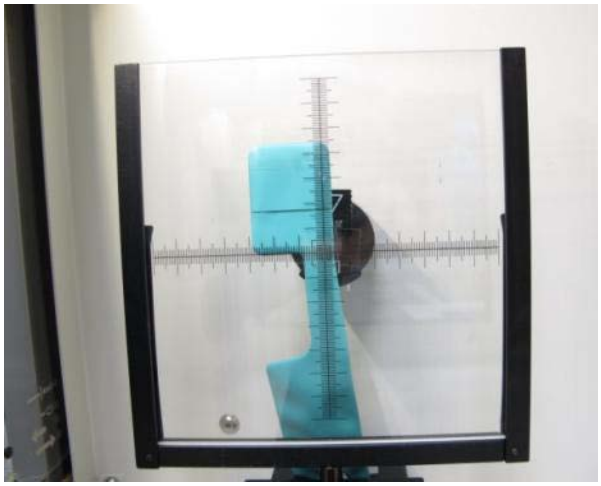
Reflection no.	y/mm	x/mm	L/mm	ϑ_{exp}/°	$h\ k\ l$	ϑ_{cal}/°	$k\ /\ l$	$y/\ z$	d/pm	λ/pm
1	4.0	12.5	13.25	17.29	1 1 3	17.55	0.33	0.32	121.4	72.2
2	0	25.5	25.5	26.66	2 0 4	26.57	0	0	100.7	90.4
3	9.75	19.0	21.25	24.17	2 2 4	24.09	0.5	0.51	82.2	67.3
4	6.75	6.75	9.50	13.34	1 3 3	13.26	1	1	92.4	42.6
5	10.75	10.75	15.50	19.33	2 4 4	19.47	1	1	90.1	59.6
6	38.25	38.25	54.50	53.30	1 1 1	35.26	1	1	232.6	268.8
7	7.0	34.0	35.50	30.75	3 1 5	30.47	0.2	0.2	68.1	69.6
8	0	45.75	45.75	33.72	4 0 6	33.69	0	0	55.8	62.0

Note

In order to keep the relative error as small as possible when determining the distances between the reflections, the following method can be applied. Transfer the diagram to transparent paper and magnify it twice with the aid of a photocopier. As an option, it is also possible to scan the pattern and to magnify it on the computer.

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 (09057-20) in combination with the XR 4.0 expert unit. If a Cu X-ray tube is used, the photography only takes 12.5 minutes and, with molybdenum tubes, good results can be achieved after just 5 minutes. The development itself takes only 2 to 3 minutes.

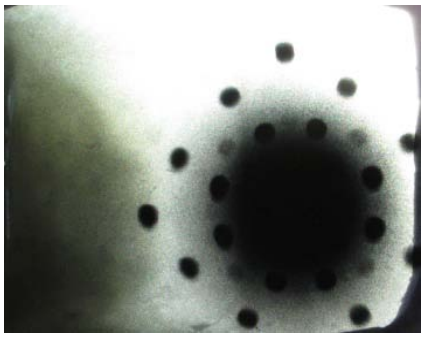


Set-up in the X-ray unit

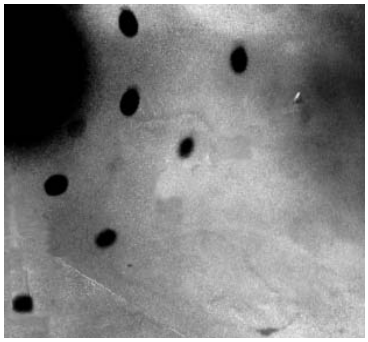
Data

Cu X-ray plug-in unit	09057-50
Tube voltage:	35 kV
Beam current:	1 mA
Diaphragm:	1 mm (09057-01)
Exposure time:	10-30 minutes

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



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

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.