Exp # 8 PH212 Jan-2019

X-ray investigation of crystal structures / Laue method 5.4.16-00

What you can learn about ...

- Crystal lattices
- Crystal systems
- Crystal classes
- Bravais lattice
- Reciprocal lattice
- Miller indices
- Structure amplitude
- Atomic form factor
- The Bragg equation

Principle:

A monocrystal is to be irradiated by a polychromatic X-ray beam and the resulting diffraction patterns recorded on film and evaluated.

What you need:

X-ray basic unit, 35 kV	09058.99	1
Plug-in module with Mo-X-ray tube	09058.60	1
Lithium fluorid crystal, mounted	09056.05	1
Crystal holder for lane diffraction	09058.11	1
Film holder	09058.08	1
Vernier caliper, plastic	03011.00	1
X-ray films, wet chemical, 100 x 100 mm, 100 pieces	09058.23	1
Bag for x-ray films, 10 pieces	09058.22	1
X-ray film developer, for 4.5 l solution	06696.20	1
X-ray film fixing, for 4.5 l solution	06696.30	1
Tray (PP), 180 x 240 mm, white	47481.00	3

Complete Equipment Set, Manual on CD-ROM included

X-ray investigation of crystal structures / Laue method P2541600

Laue pattern of an LiF (100) crystal.

Cu X-ray tube: U_A = 35 kV; I_A = 1 mA
Distance between sample and film: D = 19 mm
Exposure time: t = 120 min

Tasks:

1. The Laue diffraction of an LiF monocrystal is to be recorded on a film.
2. The Miller indices of the corresponding crystal surfaces are to be assigned to the Laue reflections.

Related topics
Crystal lattices, crystal systems, crystal classes, Bravais lattice, reciprocal lattice, Miller indices, structure amplitude, atomic form factor, the Bragg equation.

Principle
A monocrystal is to be irradiated by a polychromatic X-ray beam and the resulting diffraction patterns recorded on film and evaluated.

Equipment

X-ray basic unit, 35 kV	09058.99	1
Plug-in module with Mo X-ray tube	09058.60	1
Lithium fluoride monocrystal, mounted	09056.05	1
Crystal holder for Laue diffraction	09058.11	1
Film holder	09058.08	1
Vernier caliper, plastic	03014.00	1
Polaroid film, (ISO 3000), (9 x 12) cm, 20 sheets	09058.20	1
Polaroid film adapter	09058.21	1
or		
X-ray-film, (90 x 120) mm, 10 sheets	06696.03	1
X-ray-film developer for 4.5 l	06696.20	1
X-ray-film fixing for 4.5 l	06696.30	1
Laboratory tray, PP, 18 x 24 cm	47481.00	3

Tasks
1. The Laue diffraction of an LiF monocrystal is to be recorded on a film.
2. The Miller indices of the corresponding crystal surfaces are to be assigned to the Laue reflections.

Set-up and procedure
Fix the diaphragm tube with 1 mm diameter aperture in the X-ray tube outlet. Attach the mounted LiF-crystal with his two pins in the holder for Laue diffraction, so that the rounded side of the crystal mounting is always towards the source of X-rays. Put then this holder over the diaphragm tube. Stand the X-ray film in its light-proof envelope in the film holder at a distance of about 1.5 cm to 2 cm from the crystal. In order to obtain undistorted Laue patterns, ensure that the crystal surface and the flat film surface are parallel to each other, and that both are perpendicular to the primary beam. Expose the film at maximum values of anode voltage and anode current. An exposure time of 30 minutes is sufficient for intense reflections. If weaker reflections are also to be made visible, an exposure time of 60 minutes or longer is recommended. Measure the distance between the crystal and the film exactly for the later determination of the diffraction directions.

Theory and evaluation
Laue photographs are obtained by irradiating monocrystals with polychromatic X-rays. This method is mainly used to determine crystal symmetries and crystallographic orientations. An evaluation can be made of Laue reflection patterns from simple structures. It is, however, difficult as a rule due to the fact that the indices of the reflecting lattice planes and the wavelengths are not known.

Fig. 1: Experimental set-up for Laue photographs

Fig. 2 shows the Laue reflection pattern from an LiF monocrystal which has a face centred cubic lattice structure (fcc). If the pattern is rotated by 90° around the direction of the primary beam, it is again coherent with itself. We thus have here a fourfold symmetry, with coincidence between the direction of the beam and the crystallographic (100) direction. The intensity of the reflections depends both upon crystallographic characteristics and on the spectral intensity distribution of the X-rays.

The condition of constructive interference is determined by Bragg's relation:

$$2d \sin \vartheta = n \lambda \; ; \; (n = 1, 2, 3, \ldots) \qquad (1)$$

where:
d = the distance between the lattice planes
ϑ = the Bragg angle (glancing angle)
λ = the wavelength

For a cubic crystal with the lattice constant a, the following is valid for the spacing $d(h,k,l)$ between the lattice planes:

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

If L is the distance between a reflection and the centre of the diffraction pattern, and D the distance between sample and film (Fig. 3), then the experimentally determined glancing angle ϑ_{exp} is:

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

where y and z are the coordinates of a reflection measured from the centre of the pattern.
If the X-ray beam which coincides with the crystallographic direction [$h^*k^*l^*$] (which is here [100]) impinges on a crystal plane (h,k,l) (see Fig. 4), then the angle of incidence α is determined by the scalar product of the normal vector of the plane and the incident vector.
The following is valid for glancing angle ϑ_{cal}:
$\vartheta_{cal} = 90° - \alpha$, with

$$\cos \alpha = \frac{h\,h^* + kk^* + ll^*}{\sqrt{(h^2 + k^2 + l^2) \cdot ((h^*)^2 + (k^*)^2 + (l^*)^2)}} \qquad (4)$$

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

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

The glancing angle is calculated from (5) for all (h,k,l) triplets of planes with low indices. The angle ϑ_{exp} is determined using (3). The assignment of the single reflections to the corresponding lattice planes is found when:

$$\vartheta_{exp} = \vartheta_{cal} \qquad (6)$$

In addition to (6), the relation $k/l = y/z$ must also be valid, where z and y are the coordinates of the reflection measured from the centre of the pattern.

Fig. 2: Laue pattern of an LiF (100) crystal.
Mo-X-ray tube: U_A = 35 kV; I_A = 1 mA
Distance between sample and film: D = 19 mm
Exposure time: t = 120 min

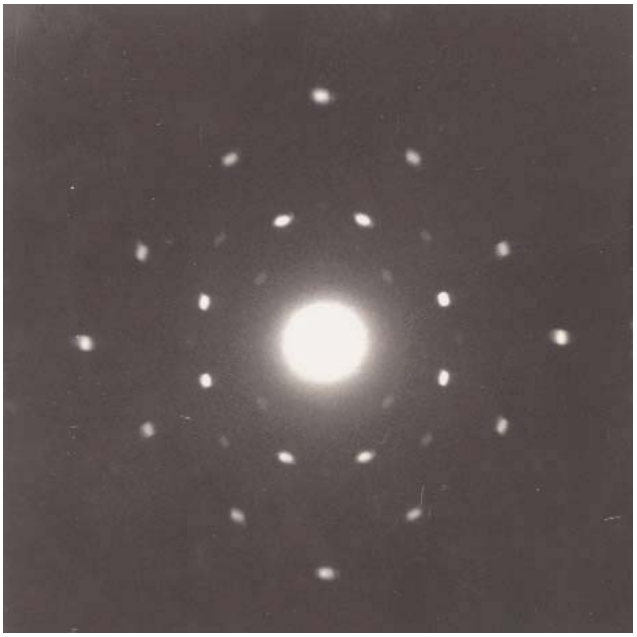
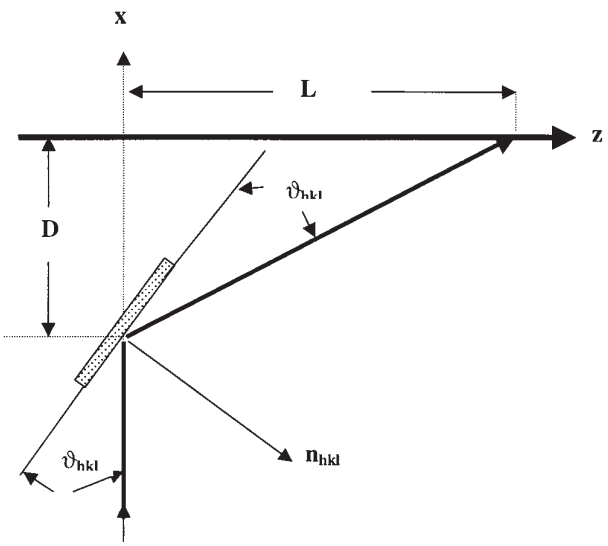


Fig. 3: Scattering geometry of the Laue method.
The Y axis lies in the plane of the film and is perpendicular to the X, Z plane.

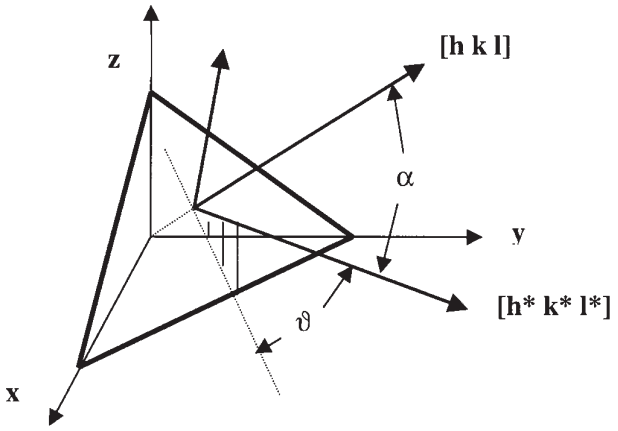


A final control can be performed as follows: The wavelengths must fulfil the condition $\lambda > \lambda_{min}$, where λ_{min} is the starting point of the bremsstrahlung, because X-ray intensity is only available for $\lambda > \lambda_{min}$. The following is valid for λ_{min} (Duane-Hunt displacement law, see Experiment P2540900):

$$\lambda_{min} = 1.24 \cdot 10^{-6}/U_A \text{ [m]} = 35.5 \text{ pm (with } U_A = 35 \text{ kV)}.$$

The Laue spots are again represented, and numbered, in Fig. 5. Due to the symmetry of the reflection pattern, evaluation can be restricted to 1/8 of the reflections. The indices of all other reflections are obtained by permutation of the (h,k,l) triplets and change of the sign. Table 1 shows the result of indexing the Laue spots.

Fig. 4: Reflection from a lattice plane with random orientation.



It results from Table 1 that reflections are visible only if the Miller indices are either all odd or all even. This is the case for a face centred cubic lattice (see Experiment P2541400).

Note

In order to keep the relative error as small as possible when determining the distances between reflections, a two-fold magnification of the reflection pattern is recommended. This can be achieved by transferring the pattern to transparent paper and magnifying this in a photocopier.

Fig. 5: Schematic representation of Laue reflections. The spots nos. 4 to 8 are very weak and can only be observed after a long exposure time.

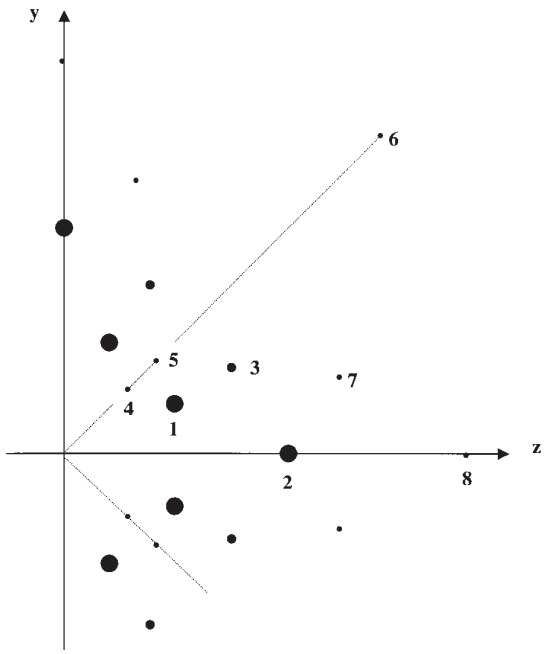


Table 1: Evaluation and results from the pattern in Fig. 2.

Spot 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	13.2_5	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	9.7_5	19.0	21.2_5	24.17	2 2 4	24.09	0.5	0.51	82.2	67.3
4	6.7_5	6.7_5	9.50	13.34	1 3 3	13.26	1	1	92.4	42.6
5	10.7_5	10.7_5	15.50	19.33	2 4 4	19.47	1	1	90.1	59.6
6	38.2_5	38.2_5	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	45.7_5	45.7_5	33.72	4 0 6	33.69	0	0	55.8	62.0