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

Principle and task
A monocrystal is to be irradiated by a polychromatic X-ray beam and the resulting diffraction patterns recorded on film and 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 package XRS 4.0 X-ray structural analysis.

Fig. 1: X-ray expert unit 09057-99
Tasks

1. Record the Laue diffraction of a LiF monocrystal on a film.
2. Assign the Miller indices of the corresponding crystal surfaces 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.

Put the film in a darkened room into the film holder and close it accurately. Position it at a distance of 15-20 mm from the sample (see Fig. 2). 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.

Ensure that the “Tube or lens side” inscription is on the proper side. Expose the film at maximum values of anode voltage and anode current. An exposure time of 15-30 min is sufficient using the tungsten tube. Set the exposure time as follows:

- Select the parameters for the X-ray tube under the item „X-ray-Parameters“ and confirm with „Enter“.
- Select „Menu“ → „Timer“ (Fig. 4) → „Duration“ and select the desired exposure time. Confirm with „enter“.
- The window „Mode“ appears, select „on“ and confirm with „enter“ (Fig. 4).
- To start the experiment lock the door and press the button under „Start“ (Fig. 5).

The irradiation is started and will be stopped automatically until time is up.

When using an X-ray film, develop it in a dark room, taking heed of the notice on the packaging. Water the film, fix it for 10 minutes, then re-water it for 10 minutes and air-dry it.
Theory

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.

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

\[ 2d \sin \vartheta = n \lambda ; \quad (n = 1, 2, 3, \ldots) \]

Where

- \( d \) = the distance between the lattice planes
- \( \vartheta \) = the Bragg angle (glancing angle)
- \( \lambda \) = the wavelength
- \( n \) = the order of diffraction

The LiF sample consists of a cubic crystal with the lattice constant \( a \).

The following is valid for the spacing \( d(hkl) \) between the lattice planes of cubic systems:

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

If \( L \) is the distance between a reflection and the centre of the diffraction pattern, and \( D \) the distance between sample and film (Fig. 6), then the experimentally determined glancing angle \( \vartheta_{\text{exp}} \) is:

\[ \vartheta_{\text{exp}} = \frac{1}{2} \arctan \frac{L}{D}; \quad L = \sqrt{y^2 + z^2} \]  

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. 7), then the angle of incidence \( \alpha \) is determined by the scalar product of the normal vector of the plane and the incident vector.

The following is valid for glancing angle \( \vartheta_{\text{cal}} \):

\[ \vartheta_{\text{cal}} = 90^\circ - \alpha, \quad \text{with} \]

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

Fig. 6: Scattering geometry of the Laue method. The Y axis lies in the plane of the film and is perpendicular to the X, Z plane.

Fig. 7: Reflection from a lattice plane with random orientation.
According to the addition theorem and from \((h^*, k^*, l^*) = (100)\), it follows from (4) that:

\[
\sin \vartheta = \frac{h}{\sqrt{h^2 + k^2 + l^2}} \tag{5}
\]

**Task 1: Record the Laue diffraction of a LiF monocrystal on a film.**

Fig. 8 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.

**Task 2: Assign the Miller indices of the corresponding crystal surfaces to the Laue reflections.**

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

\[
\vartheta_{\text{exp}} = \vartheta_{\text{cal}} \tag{6}
\]

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.

A final control can be performed as follows. The lattice constant of LiF is \(a = 402.8\) pm. The distances of the lattice planes can first be calculated with this value and using (2), and then the corresponding wavelengths using (1). These wavelengths must fulfill the condition \(\lambda > \lambda_{\text{min}}\), where \(\lambda_{\text{min}}\) is the starting point of the bremsstrahlung, because X-ray intensity is only available for \(\lambda > \lambda_{\text{min}}\). The following is valid for \(\lambda_{\text{min}}\) (Duane-Hunt displacement law, see Experiment 5.4.09):

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

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

W X-ray tube: \(U_A = 35\ kV; I_A = 1\ mA\)

Distance between sample and film: \(D = 15\ mm\)

Exposure time: \(t = 15\ min\)

Fig. 9: Schematic representation of Laue reflections.

The spots nos. 4 to 8 are very weak and can only be observed after a long exposure time.
The Laue spots are again represented, and numbered, in Fig. 9. 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.

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 2541401).

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

<table>
<thead>
<tr>
<th>Spot no.</th>
<th>(y/\text{mm})</th>
<th>(x/\text{mm})</th>
<th>(L/\text{mm})</th>
<th>(\theta_{\text{exp}}^\circ)</th>
<th>(h\ k\ l)</th>
<th>(\theta_{\text{cal}}^\circ)</th>
<th>(k/\ell)</th>
<th>(y/l)</th>
<th>(d/\text{pm})</th>
<th>(\lambda/\text{pm})</th>
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<tr>
<td>1</td>
<td>4.0</td>
<td>12.5</td>
<td>13.25</td>
<td>17.29</td>
<td>1 1 3</td>
<td>17.55</td>
<td>0.33</td>
<td>0.32</td>
<td>121.4</td>
<td>72.2</td>
</tr>
<tr>
<td>2</td>
<td>0</td>
<td>25.5</td>
<td>25.5</td>
<td>26.66</td>
<td>2 0 4</td>
<td>26.57</td>
<td>0</td>
<td>0</td>
<td>100.7</td>
<td>90.4</td>
</tr>
<tr>
<td>3</td>
<td>9.75</td>
<td>19.0</td>
<td>21.25</td>
<td>24.17</td>
<td>2 2 4</td>
<td>24.09</td>
<td>0.5</td>
<td>0.51</td>
<td>82.2</td>
<td>67.3</td>
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<tr>
<td>4</td>
<td>6.75</td>
<td>6.75</td>
<td>9.50</td>
<td>13.34</td>
<td>1 3 3</td>
<td>13.26</td>
<td>1</td>
<td>1</td>
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</tr>
<tr>
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<td>10.75</td>
<td>15.50</td>
<td>19.33</td>
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<td>19.47</td>
<td>1</td>
<td>1</td>
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<td>1</td>
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<tr>
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<td>35.50</td>
<td>30.75</td>
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<td>30.47</td>
<td>0.2</td>
<td>0.2</td>
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<tr>
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<td>33.72</td>
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<td>0</td>
<td>0</td>
<td>55.8</td>
<td>62.0</td>
</tr>
</tbody>
</table>

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.
X-ray investigation of crystal structures / Laue method

TEP
5.4.16-01