Debye-Scherrer photography: determining the lattice plane spacings of polycrystalline powder samples

**Objects of the experiment**
- Evaluating Debye-Scherrer photographs of an NaCl and an LiF sample.
- Investigating the lattice structure of NaCl and LiF crystals.
- Determining the lattice constants and the lattice plane spacings.

**Principles**

**Debye-Scherrer photographs:**

For taking a Debye-Scherrer photograph, a powdery crystalline sample is transilluminated with monochromatic X-rays. The interference pattern of the scattered radiation is frozen on an X-ray film. The powder sample contains minute monocrystals of about 5–50 \( \mu \text{m} \) diameter, so-called crystallites. A set of lattice planes in a crystallite leads to a diffraction reflection on the X-ray film if it is aligned so that the Bragg condition

\[
n \cdot \lambda = 2 \cdot d \cdot \sin \theta \tag{I}
\]

is fulfilled (see Fig. 2 and experiment P6.3.3.1). The angle between the diffraction reflection and the film, which is aligned perpendicularly to the primary ray, is \( 2\theta \).

In general the crystallites are randomly oriented without any privileged direction so that there are always some crystallites in the crystal powder which correspond to a rotation of the crystallite under consideration around the primary axis. In the arrangement of the film selected here, their diffraction reflections form a circle on the X-ray film with the radius

\[
R = L \cdot \tan 2\theta \tag{II}
\]

where

- \( L \): distance between the sample and the film
- \( R \): radius of the diffraction circle

The finer the powder is, the more uniformly the individual reflections of the crystallites will be lined up to form a circle.

The complete diffraction pattern is a set of concentric circles. Because of Eqs. (I) and (II), each radius \( R \) corresponds to a certain lattice plane spacing \( d \) and a certain diffraction order \( n \) or, more precisely, a certain ratio \( \frac{d}{n} \).

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**Fig. 1** Scheme of the setup for taking Debye-Scherrer photographs
- a X-ray tube
- b Zr filter
- c collimator
- d Sample
- e X-ray film

**Fig. 2** Bragg reflection at an “appropriate” set of lattice planes of a particular crystallite in the powder sample
1: collimator,
2: set of lattice planes,
3: film

\[ R = L \cdot \tan 2\theta \]
If the consideration is restricted to cubic crystals, the lattice plane spacing can be expressed in the form

\[ d = \frac{a_0}{\sqrt{h^2 + k^2 + l^2}} \]  

(III);

\( a_0 \): lattice constant

Here the integers \( h, k, l \) are the Miller indices of the set of lattice planes under consideration (see experiment P7.1.2.2). If (III) is inserted into Eq. (I), the quadratic form

\[ \sin^2 \theta = \left( \frac{\lambda}{2a_0} \right)^2 \cdot \left( (n \cdot h)^2 + (n \cdot k)^2 + (n \cdot l)^2 \right) \]  

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is obtained.
The fine graininess with a granule diameter below 10 μm is centred on the pinhole diaphragm in the primary ray. A 0.1 to 0.5 mm thickness is an appropriate sample. This sample consists of two pieces of transparent adhesive tape in a layer of approx. 300 μm. A small quantity of fine powder which is embedded between the tapes is also included; this results in a reference photograph with a coarse-grained metal powder.

A short calculation shows that

\[ A = 4 \cdot f_A - 4 \cdot f_H, \quad \text{if } h, k, l \text{ odd} \]

\[ A = 4 \cdot f_A + 4 \cdot f_H, \quad \text{if } h, k, l \text{ even} \]

The amplitudes \( A \) of the waves starting from the unit cells thus only are different in zero if all indices \( h, k, l \) are even or if they are all odd. A combination of even indices leads to a greater amplitude \( A \) than a combination of odd indices. For other crystal structures other selection rules apply.

**Evaluating a Debye-Scherrer photograph:**

In this experiment, Debye-Scherrer photographs of crystals with NaCl structure are taken. The Bragg angles in the photograph are obtained according to Eq. (II) from the radii \( R \) of the diffraction rings and the distance \( L \) between the sample and the film. For the evaluation, the associated values of \( \sin^2 \theta \) are decomposed into a constant factor \( F \) and the smallest integer \( Z \) (see Eq. (V)) whose Miller indices \( h, k, l \) fulfill the selection rules (XI).

From the mean value of the factors \( F \) obtained from the Debye-Scherrer photograph and the wavelength of the molybdenum \( K_\alpha \) radiation (\( \lambda = 71.1 \text{ pm} \)) the lattice constant \( a_0 \) can be calculated by applying Eq. (VI). Then the lattice plane spacings \( d \) are derived according to Eq. (III).

**Setup and carrying out the experiment**

The experimental setup is illustrated in Fig. 4.

- If necessary, remove the goniometer or the plate capacitor (X-ray).
- Dismount the collimator, mount the Zr filter (a) (from the scope of supply of the X-ray apparatus) on the ray entrance side of the collimator and re-insert the collimator.

a) **Debye-Scherrer photograph of NaCl:**

- Carefully grind the dry NaCl salt in the mortar, and embed an approx. 0.4 mm thick layer between two pieces of transparent adhesive tape.
- Carefully attach the sample (c) to the pinhole diaphragm (b) with adhesive tape (from the scope of supply of the film holder X-ray), and put the pinhole diaphragm onto the collimator.
- Clamp the X-ray film (d) at the film holder so that it is centred on the marked area, and see to it that the entire surface of the film is planar.
- Clamp the film holder onto the experiment rail, and mount the experiment rail in the experiment chamber of the X-ray apparatus.
- Make a 13 mm long spacer from paper board and shift the film holder so that the distance between the sample and the film is 13 mm (by varying the distance between the sample and the film the area covered in the photograph is changed).
- Set the tube high voltage \( U = 35 \text{ kV} \), the emission current \( I = 1.0 \text{ mA} \) and \( \Delta \theta = 0.0^\circ \).
- Select the measuring time \( \Delta t = 14400 \text{ s} \), and start the exposure timer with the key Scan.

If the exposure time is longer, the reflections near the centre are blurred by the unscattered X-rays; however structures which are farer away from the centre become discernable.

- When the exposure time is over, take the film holder with the experiment rail out of the experiment chamber.
- Remove the X-ray film from the holder, and develop it according to the instruction sheet for the X-ray film.

b) Debye-Scherrer photograph of LiF:
- Exchange the NaCl ample with the carefully ground LiF sample.
- Clamp a new X-ray film in the film holder, and mount the experiment rail with the film holder once more.
- Start the exposure timer with the key Scan.
- When the exposure time is over, take the X-ray film from the film holder and develop it.

Measuring example
a) Debye-Scherrer photograph of NaCl: (see Fig. 5)
b) Debye-Scherrer photograph of LiF: (see Fig. 6)

Evaluation
- Determine the diameter D of the diffraction rings with the precision vernier calliper.
- Calculate the Bragg angle \( \theta \) using Eq. (II) to obtain \( \sin^2 \theta \).
- “Guess” the integer factor \( Z \), and use Eq. (V) to calculate the factor F.

\[
\begin{array}{cccccccc}
\text{Nr.} & \text{D mm} & \theta & \sin^2 \theta & n & h & k & l & Z & F \\
1 & 10.0 & 10.5 & 0.033 & 1 & 2 & 2 & 0 & 8 & 0.0042 \\
2 & 12.5 & 12.8 & 0.049 & 1 & 2 & 2 & 2 & 12 & 0.0041 \\
3* & 14.5 & 14.6 & 0.063 & 1 & 4 & 0 & 0 & 16 & 0.0040 \\
4 & 17.0 & 16.6 & 0.082 & 1 & 4 & 2 & 0 & 20 & 0.0041 \\
5 & 19.3 & 18.3 & 0.099 & 1 & 4 & 2 & 2 & 24 & 0.0041 \\
6* & 23.0 & 20.7 & 0.125 & 1 & 4 & 0 & 0 & 32 & 0.0039 \\
7 & 25.0 & 21.9 & 0.139 & 1 & 6 & 4 & 0 & 36 & 0.0039 \\
8 & 28.0 & 23.6 & 0.160 & 1 & 6 & 2 & 0 & 40 & 0.0040 \\
\end{array}
\]

* only weak

In Table 1, the decomposition of the experimental results for \( \sin^2 \theta \) into the factors F and Z and the associated Miller indices h, k, l and the diffraction order n are listed. The mean value of...
the factors $F$ is 0.00403. From this the lattice constant of NaCl is calculated:

$$a_0 = \frac{\lambda}{2} \cdot \frac{1}{\sqrt{F}} = 560 \text{ pm}$$

Value quoted in the literature [1]: $a_0 = 564.02 \text{ pm}$

In Table 2 the lattice plane spacings calculated from the literature value of $a_0$ and Eq. (III) are given.

Tab. 2: Lattice plane spacings $d$ contributing to the Debye-Scherrer photograph of NaCl

<table>
<thead>
<tr>
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<th>k</th>
<th>l</th>
<th>$d_{\text{mm}}$</th>
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b) Debye-Scherrer photograph of LiF:

In Table 3, the decomposition of the experimental values for $\sin^2 \theta$ into the factors $F$ and $Z$ and the associated Miller indices $h, k, l$ and the diffraction order $n$ are listed. The mean value of the factors $F$ is 0.00767. From this the lattice constant of LiF is calculated:

$$a_0 = \frac{\lambda}{2} \cdot \frac{1}{\sqrt{F}} = 406 \text{ pm}$$

Value quoted in the literature [1]: $a_0 = 402.8 \text{ pm}$

In Table 4 the lattice plane spacings calculated from the literature value of $a_0$ and Eq. (III) are given.

Tab. 4: Lattice plane spacings $d$ contributing to the Debye-Scherrer photograph of LiF

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<thead>
<tr>
<th>Nr.</th>
<th>h</th>
<th>k</th>
<th>l</th>
<th>$d_{\text{mm}}$</th>
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<td>82</td>
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</table>

Results

A Debye-Scherrer photograph is a diffraction photograph of a powder sample with monochromatic X-rays.

In a plane perpendicular to the primary ray, the individual reflections of the crystallites form a system of concentric diffraction rings. These are denser and more uniform the finer the crystal powder is. Intensity maxima that occur on the rings originate from larger crystals which may be due to insufficient grinding.

From the selection rules related to the sets of lattice planes, conclusions regarding the crystal structure can be drawn.

Literature
