X-ray investigation of crystal structures / Laue method with digital X-ray detector (XRIS) (Item No.: P2541602)

Curricular Relevance

Area of Expertise: Physics → **Education Level:** University → **Topic:** Modern Physics → **Subtopic:** X-ray Physics → **Experiment:** X-ray investigation of crystal structures / Laue method with digital X-ray detector (XRIS)

Difficulty	Preparation Time	Execution Time	Recommended Group Size
Difficult	1 Hour	2 Hours	2 Students

Additional Requirements:

- PC

Experiment Variations:

Keywords:

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

Overview

Short description

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 pat-tern is photographed and then evaluated. A monocrystal X-ray structure analysis can be performed live during a lecture with the aid of the XR 4.0 expert unit and the direct digital x-ray image sensor. If a Cu X-ray tube is used, the photography only takes 1 minute.

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Fig. 1: P2541602

Note: This experiment can also be performed with a copper or molybdenum X-ray tube.

Equipment

Position No.	Material	Order No.	Quantity
1	XR 4.0 expert unit, X-ray unit, 35 kV	09057-99	1
2	XRCT 4.0 X-ray Computed Tomography upgrade set	09180-88	1
3	XR 4.0 X-ray plug-in unit W tube	09057-81	1
4	XR 4.0 X-ray LiF crystal, mounted	09056-05	1
5	XR 4.0 X-ray Crystal holder for Laue-pattern	09058-11	1
6	Vernier calliper stainless steel 0-160 mm, 1/20	03010-00	1
7	XR 4.0 X-ray optical bench	09057-18	1
8	XR 4.0 X-ray Diaphragm tube d = 1 mm	09057-01	1

Tasks

1. Take a photograph of the Laue pattern of a LiF monocrystal performing the following steps

a) Take an offset-Image.

b) Take the Image of the Laue pattern.

c) Process Image.

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

Setup and procedure

Setup

- Install the 1 mm collimator and place the Crystal holder for Laue diffraction on the diaphragm (Fig. 2).

Fig. 2: Installation of diaphragm and crystal holder.

- Attach the XRIS to its holder.
- Place the Digital X-ray detector XRIS on the rail at position 9 cm. The back side of the XRIS stage corresponds to its position on the rail (Fig. 3).

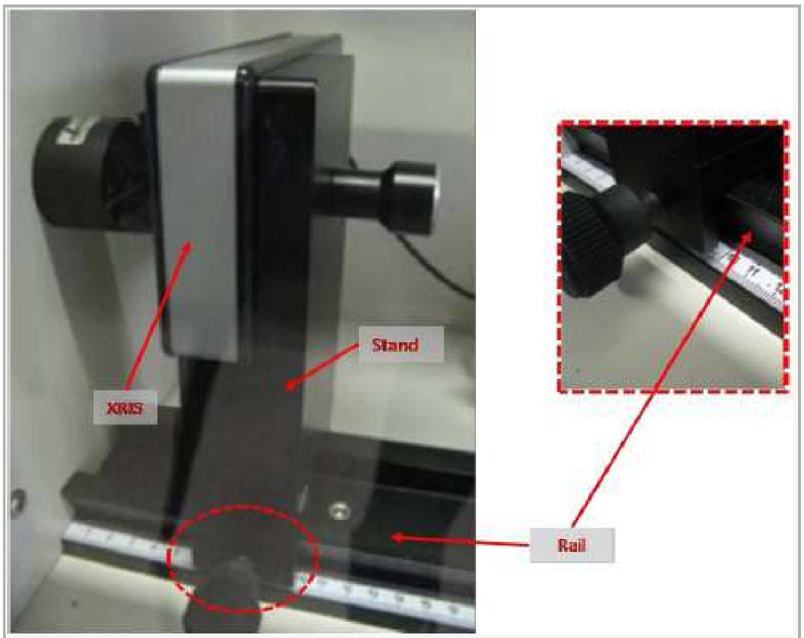

Fig. 3: XRIS setup

- Connect the X-ray unit via USB cable to the USB port of your computer (the correct port of the X-ray unit is marked in Fig. 4).

Fig. 4: Commection of the computer

- Connect the USB cable of the detector to the computer
- Start the “measureCT” program. A virtual X-ray unit, rotation stage and detector will be dis-played on the screen. The green indication LED on the left of each components indicates that its presence has been detected (Fig. 5)

- You can change the High Voltage and current of the X-ray tube in the corresponding input windows or manually on the unit. (Fig. 5)
- When clicking on the unit pictogram additional information concerning the unit can be retrieved (Fig. 5)
- The status pictogram indicates the status of the unit and can also be used to control the unit such as switching on and off the light or the X-rays (Fig. 5)

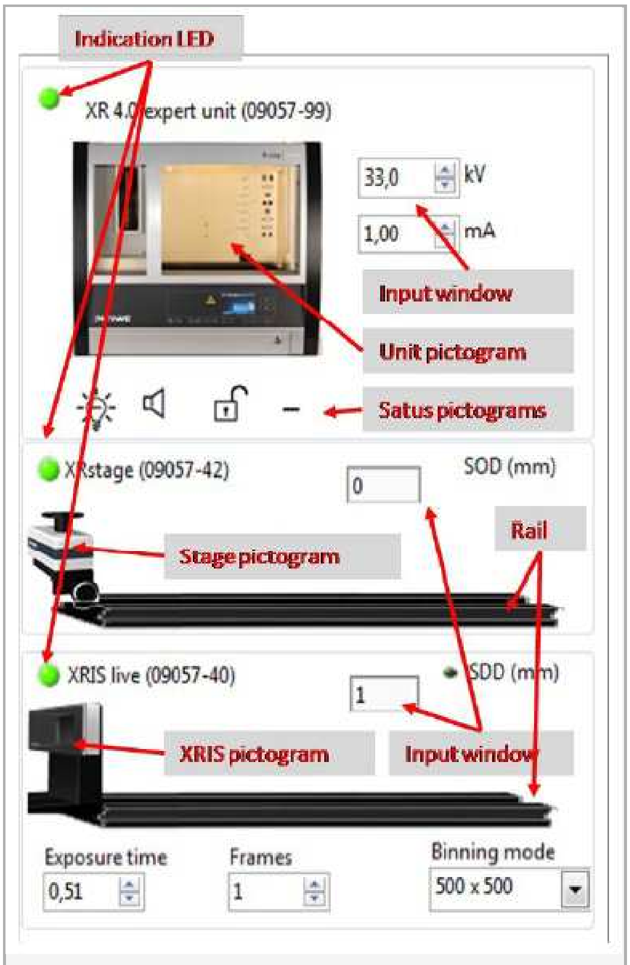

Fig. 5: Part of the user interface of the software

- The settings of the XRIS can be adjusted using the input windows.

  o The **exposure time** controls the time between two frames are retrieved from the detector
  o the **number of frames** defines how many frames are averaged
  o and with the **binning mode** the charge of neighbouring pixels is averaged to reduce the total amount of pixels in one frame.

Procedure

Experiment execution

1a) Take an offset-Image.

- Start a new experiment, give it a unique name and fill in your details (Fig. 6). Alternatively it is also possible to load this experiment with pre-recorded images. The correct configuration will be loaded automatically as well.

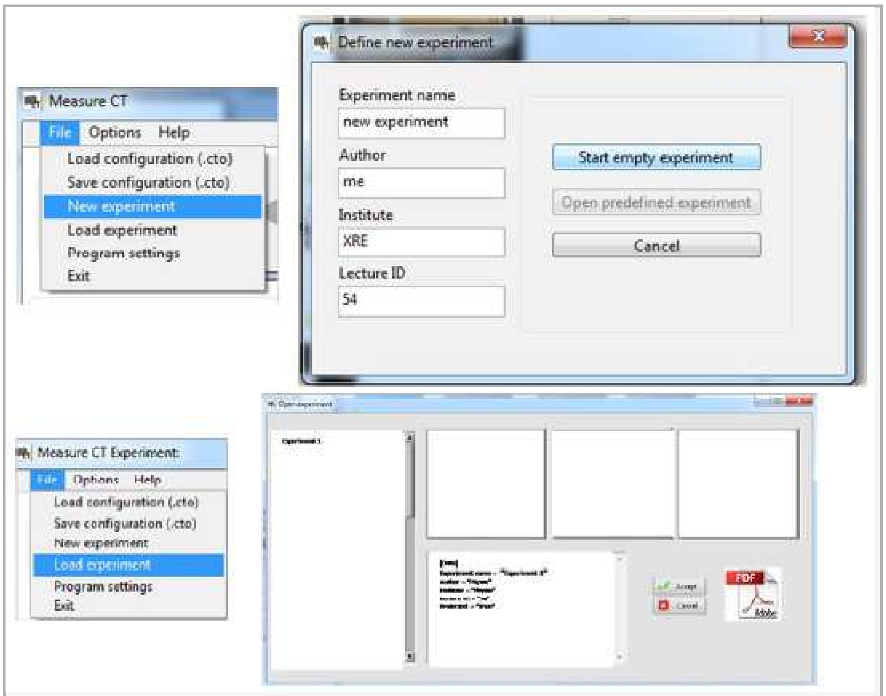

Fig. 6: Start a new experiment and fill in your details

- Adjust the XRIS settings and X-ray unit settings according to Fig. 7 or load the configuration from the predefined CTO file 'Experiment 10' (see Fig. 7).

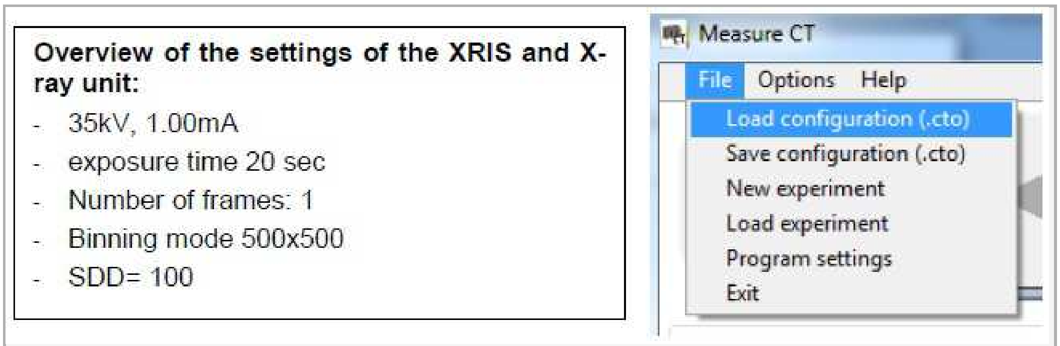

Fig. 7: The settings for this experiment (left panel) and the method load and adjust the settings (right panel)

Do not calibrate the XRIS!

- The detector is still at approx. 9 cm and the diaphragm and the Laue crystal holder are also installed.
- Activate the 'Live view' (see Fig. 8). When the Live view is activated, every new image that is retrieved from the X-ray detector is displayed and the small green light next to the XRIS live indication blinks.

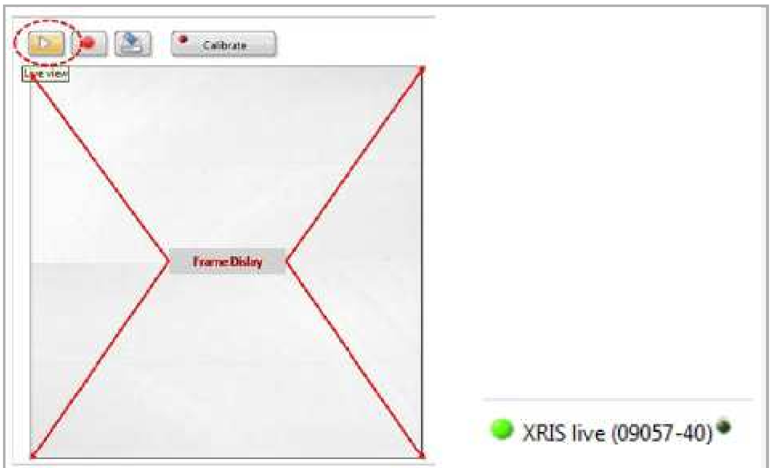

Fig. 8 'Live View' button (left panel) and blinking green light (right panel)

- Wait until at least 3 new images have been renewed in order to let the dark current of the detector stabilise (see theory for more information).
- To save the resulting image, stop the live view. When the next first image is captured, the 'Image processing' buttons become available. Click on 'Save Image'. Four formats of images can be saved (tiff, png, jpg and bmp). **Save this image as tiff and give is the name 'DI1', which stands for *Dark Image with 1 frame*. This is your Offset-Image (Fig. 9).**

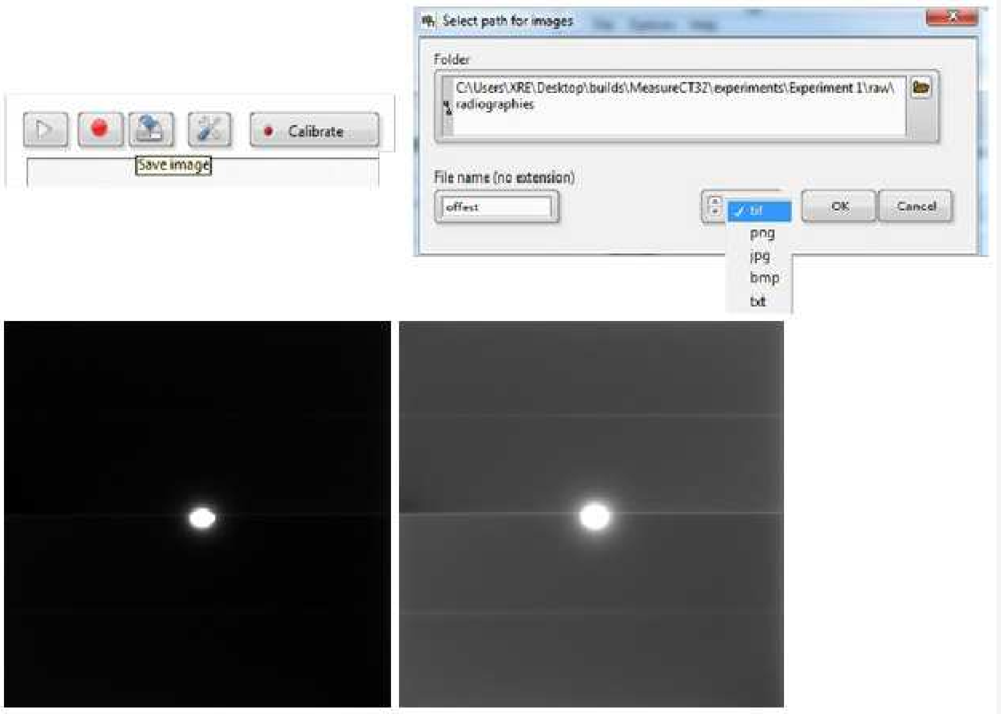

Fig. 9: Instructions how to save the image and resulting Image: Left: Tiff-Image DI1. Right: the same image with enhanced contrast and brightness.

1b) Take the Laue diffraction pattern.

Now place the LiF-crystal in the holder (Fig. 10) and move the XRIS to the same position (approx. 9 cm) as in task 1a).

Fig. 10: Place the crystal in the holder

- Switch on the X-rays, active the 'Live View' (do again not calibrate the sensor) and wait again until at least three images have been displayed.
- Stop the Live View and save the image as Laue1. You can already see the Laue diffraction pattern.

1c) Processing of the image.

- To further process an image you need to open the *Image Viewer* (Fig. 11). The Image Viewer can be opened in two ways, either from the taskbar or using the shortcut button (more information on the Image Viewer in experiment1).

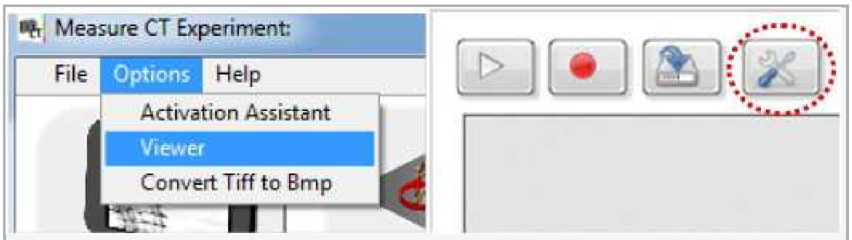

Fig. 11: Open Image Viewer instructions

- To process images that you saved in tasks 1 a) and b), open the destination directory (Fig. 12) by choosing “Image” and “Select Image path” (if you started an experiment before performing steps 1 a) and b) the right folder opens automatically).

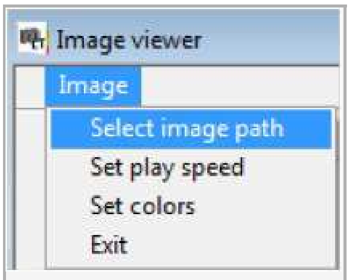

Fig. 12: Select the image path

- Double click on 'DI1' from the list, set this image as offset by clicking on “Set as offset” and select the normalisation (Fig. 13). Any new image that you select from the list will from now be subtracted with DI1.

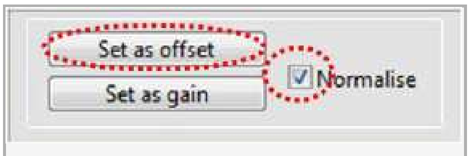

Fig. 13: Correct for offset

- Select the ' Laue1' image from the list. The displayed image is now the 'Laue1' subtracted with 'DI1'.
- Autoscale the histogram of the displayed image by clicking on “Autoscale image color” (Fig. 14).

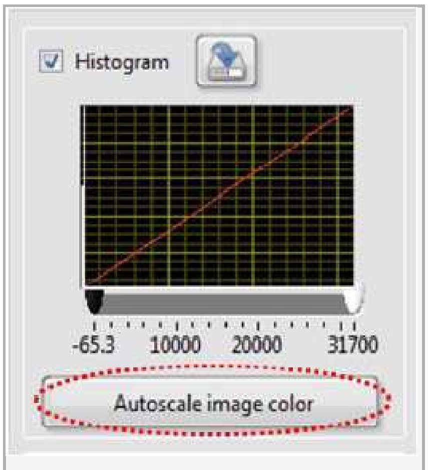

Fig. 14: Autoscale and adjust the Middle colour

- Adjust the upper contrast handls (Fig. 15) so that the Laue patterns become visible. The current image is composed of three parts.

  o The direct beam that has been collimated by the 1mm collimator. This part of the image results in the bright spot in the middle of the detector. As many X-ray photons have interacted with the detector, this direct beam will have saturated the detector.

  o The Laue pattern spots that have been caused by the interaction of the direct beam with the crystal. Only few X-ray photons are diffracted and thus, the spots are composed of few detector counts.

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o The largest part of the detector has not detected any X-ray photons.

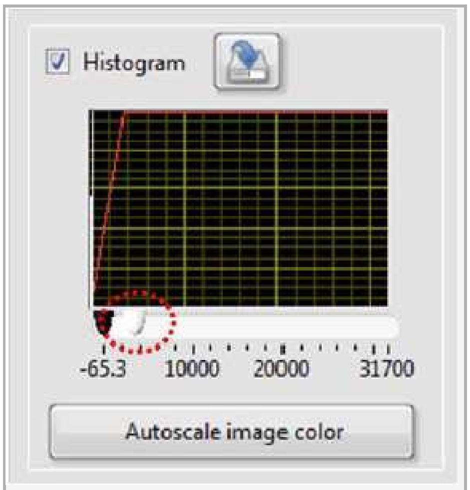

Fig. 15: Adjust the upper contrast handle to the left until the laue patterns become visible

- In order to further optimise the quality of the image the contrast of the image has to be adjusted to the laue spots. With the auto scale tool, the entire range of grey-values of the saved images are dis-played (0-30000). By adjusting the contrast handles, you can focus on the spots only. Since very few counts are registered in these spots the lower and upper contrast handles will have to be on the far left or right side of the histogram. It is possible to manually change the upper level value (Fig. 16) of the displayed histogram by typing in a new upper value (for example 2000).

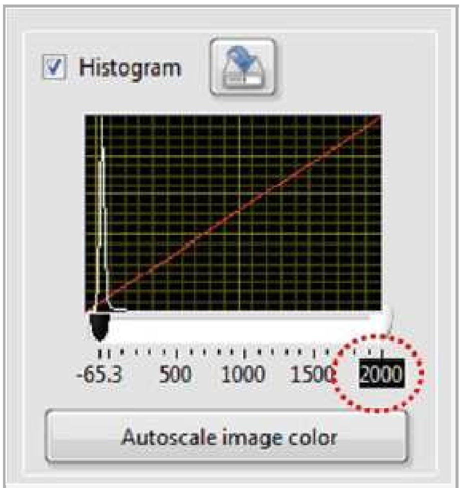

Fig. 16: Adjust the upper contrast level to a smaller value

- Save the image with adjusted contrast as a bmp, jpg or tif.

Note

To optimize the image quality repeat this experiment with different exposure times and number of frames.

Alternatively, it is also possible to vary the kV settings.

For every change in either the exposure time and the kV settings both new Offset and Laue images have to be taken and saved.

Theory and evaluation

Theory

Detector saturation

With digital X-ray imaging, X-ray photons that interact with the detector are converted to a digital signal. Such a digital detector is composed of a raster of pixels (picture elements) and each pixel can be considered as bucket. For each interaction of an X-ray photon with the detector, a series of electrons are produced in the pixel corresponding with the location of the photon interaction. These electrons are stored in the pixel, gradually filling up the bucket. After a set time interval, " ***exposure time*** *", the electron content of the pixel is measured by emptying it. For the same intensity of X-ray's, a longer exposure time will results in a larger number of pixels in the bucket.*

*Each digital detector has a limited bucket size which is called the '****full well capacity*** *' of the detector. When this level off fill is reached, additional electrons are thrown away because the detector is saturated. A saturated detector will cause inconsistent measurements and has thus to be avoided.*

Detector calibration

Each digital detector has a different and variable offset and pixel-specific output. During the calibration these variations will be measured and used in the subsequent imaging.

Even without the X-rays on, the detector will generate a read-out value that is different from 0, called 'dark image' or 'affset'. This has several reasons from which the main reasons are an electronic offset and read-out noise. When determining the beam intensity I_0 *, it is important to subtract this offset (*I_D *) from the measured read-out (*$I_{0,M}$ *).*

$$I_0 = I_{0,M} - I_D$$

Interpretation of Laue diffraction patterns

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:

$2d\sin\vartheta = n\lambda$ *; (n = 1, 2, 3,...)*

(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:*

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

IfL *is the distance between a reflection and the centre of the Laue pattern, and*D *the distance between the film and the sample (Fig. 17), then the glancing angle*ϑ_{exp} *that is determined in an experimental manner is:*

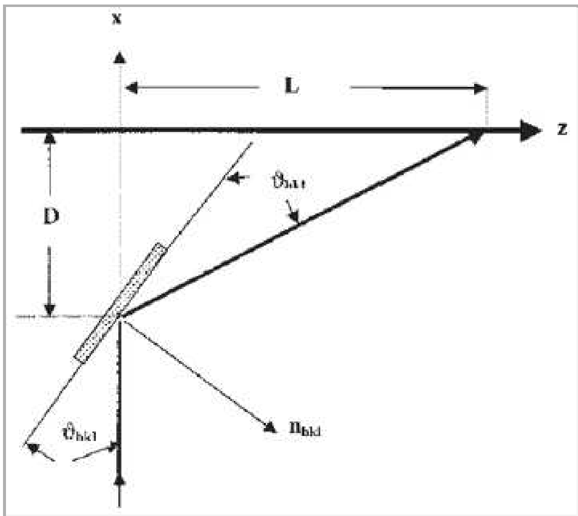

Fig. 17: Scattering geometry of a Laue pattern. The y-axis is in the plane of the film and is perpendicular to the x,z plane.

$\vartheta_{\text{exp}} = \frac{1}{2}\arctan\frac{L}{D}$ *;* $L = \sqrt{y^2 + z^2}$ *(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 crystallo-graphic 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. 18) is determined by the scalar product of the normal vector of the plane and the incident vector.*

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

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

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

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

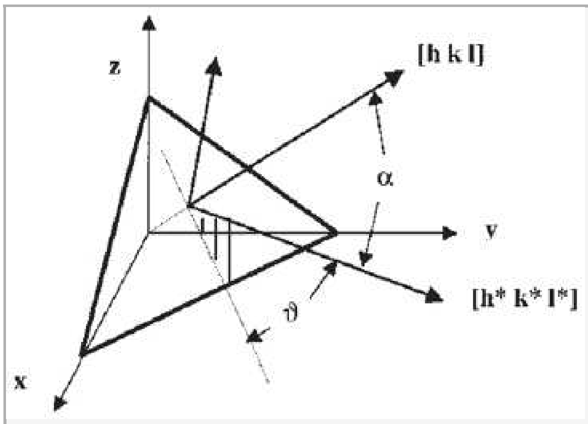

Fig. 18: Reflection on a lattice plane with random orientation.

Evaluation

Task 1: Take a photograph of the Laue pattern of a LiF monocrystal.

Figure 19 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 re-flections depends on the reflecting crystal surface as well as on the spectral intensity distribution of the X-rays.

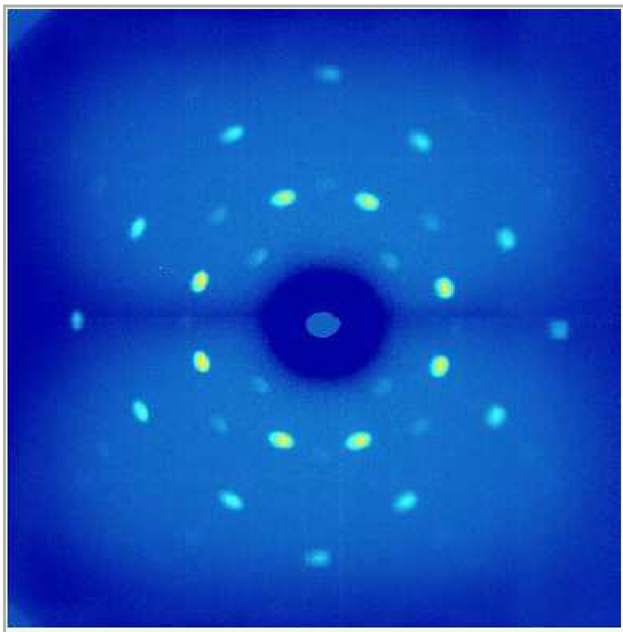

Fig. 19: Laue pattern of the LiF (100) crystal. Operating values of the tungsten X-ray tube: Accelerating voltage UA = 35 kV; anode current IA = 1 mA; exposure time 20 s, 1 frame per image, binning mode 500 x 500.

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* $\vartheta_{\exp}$ *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 $\lambda_{min} = 1.24 \cdot 10^{-6} / U_A [m]$ *. For an accelerating voltage* $U_A = 35kV$ *, the following is true:* $\lambda_{min} = 35.5pm$ *. This means that for the assignment of the reflections to the lattice planes, only X-rays with a wavelength of* $\lambda > 35.5pm$ *can play a role.*

Figure 20 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.*

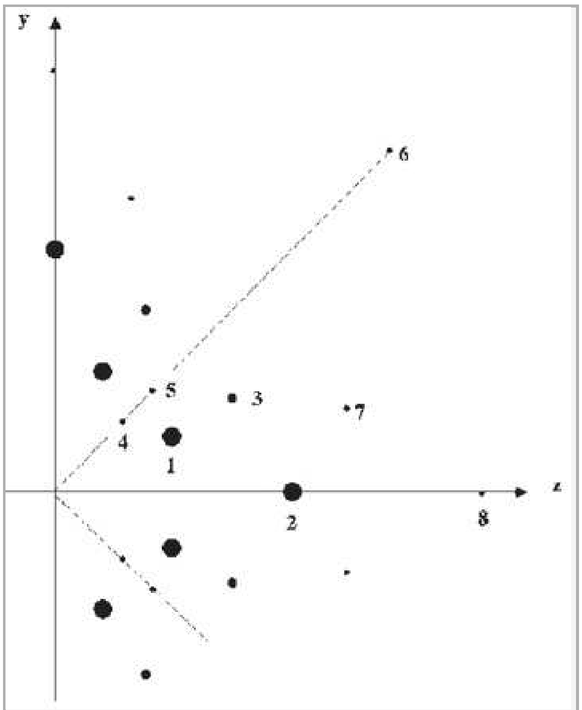

Fig. 20: Schematic representation of the Laue reflections.

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	$\vartheta_{\text{exp}}/°$	hkl	$\vartheta_{cal}/°$	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	26.75	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, magnify it on the computer and print it in enlarged form.

To shadow the primary beam spot on the Laue Image which sometimes leads to artefacts on the digital image it can be useful to use am beam stop. Just stick a small piece of metal (diameter approx. 3 mm) to a piece of tape and fix it in front of the senor right in the middle of the active area. Optimize the position by taking images (exposure time around 1 s) and checking in the live view image if the primary beam is completely covered by the peace of metal.

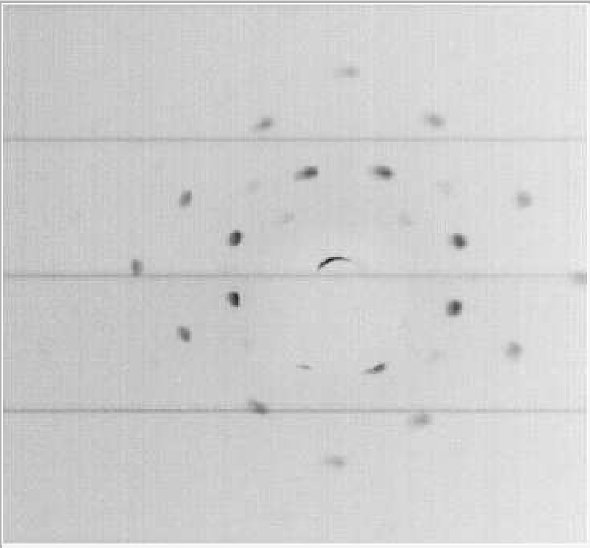

Fig 21: Laue diagram with Beamstop.