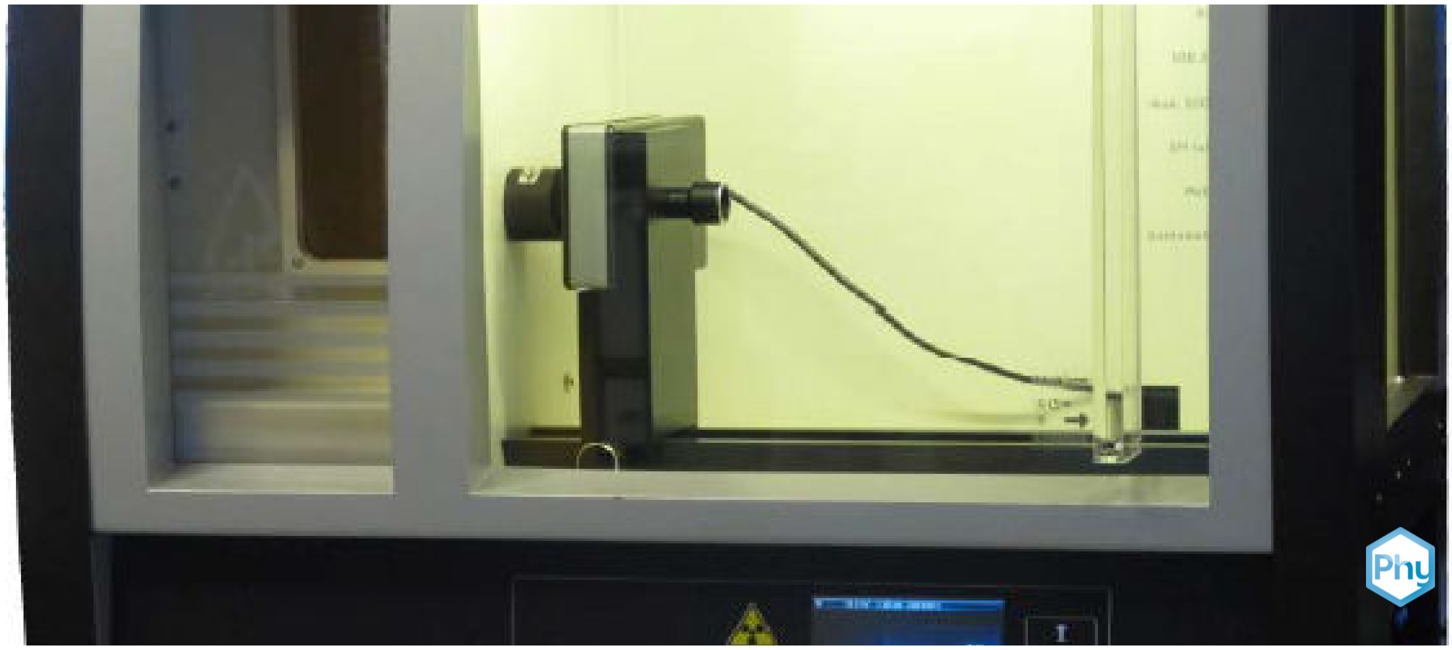
X-ray investigation of crystal structures / Laue method with digital X-ray image sensor(XRIS)



Physics | Modern Physics | Production & use of X-rays

Difficulty level	Group size	Preparation time	Execution time
hard	2	45+ minutes	45+ minutes

This content can also be found online at:

http://localhost:1337/c/5f608fa07e9d5b0003e1e809

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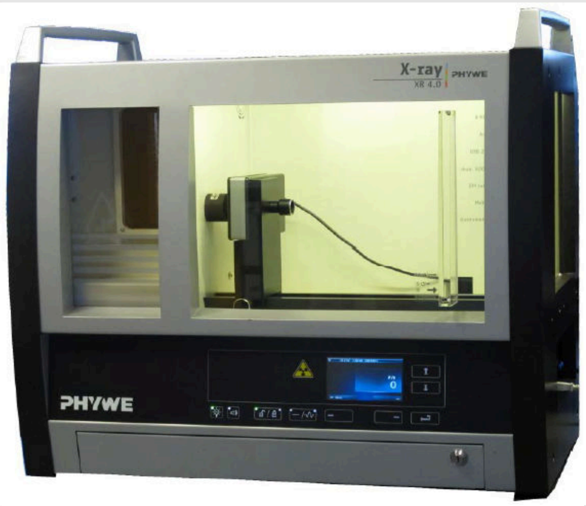
General information

Application



Setup

Most applications of X rays are based on their ability to pass through matter. Since this ability is dependent on the density of the matter, imaging of the interior of objects and even peaple becomes possible. This has wide usage in fields such as medicine or security.

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Other information (1/2)

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Prior knowledge

The prior knowledge for this experiment is found in the Theory section.

Main 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.

Other information (2/2)

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Learning objective

The goal of this experiment is to investigate the Laue pattern of a LiF monocrystal.

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.

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Theory (1/5)

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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:

$$2\mathrm{d}\sin(\theta) = \mathrm{n}\lambda \qquad (1)$$

(d = interplanar spacing; θ = glancing angle; λ = wavelength; 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:

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

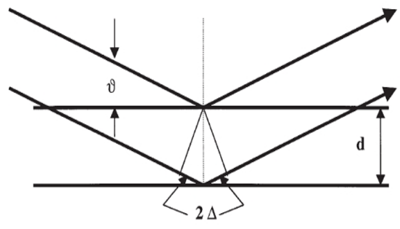

Fig. 1: Bragg scattering on a pair of lattice planes

Theory (2/5)

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

$$\theta_{\mathrm{exp}} = \frac{1}{2}\arctan\left(\frac{\mathrm{L}}{\mathrm{D}}\right);\ \mathrm{L} = \sqrt{\mathrm{y}^2+\mathrm{z}^2} \qquad (3)$$

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

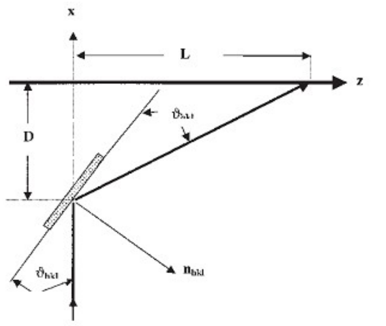

Fig. 2: Scattering geometry of a Laue pattern. The y-axis is in the plane of the film and is perpendicular to the x,z plane.

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Theory (3/5)

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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. 3) 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: $\theta_{\mathrm{cal}} = 90^\circ - \alpha$

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

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

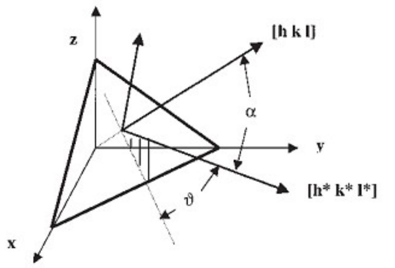

Fig. 3: Reflection on a lattice plane with random orientation.

Theory (4/5)

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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.

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Theory (5/5)

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 $\mathrm{I_0}$,it is important to subtract this offset ($\mathrm{I_D}$) from the measured read-out ($\mathrm{I_{0,M}}$).

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

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Equipment

Position	Material	Item No.	Quantity
1	XR 4.0 expert unit, 35 kV	09057-99	1
2	XR4 X-ray Plug-in Cu tube	09057-51	1
3	XR 4.0 X-ray structural analysis upgrade set	09145-88	1
4	XR 4.0 X-ray Computed Tomography upgrade set	09185-88	1

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Setup and Procedure

Setup (1/2)

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- Install the 1 mm collimator and place the Crystal holder for Laue diffraction on the diaphragm (Fig. 4).
- 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. 5).

Fig. 4: Installation of diaphragm and crystal holder.

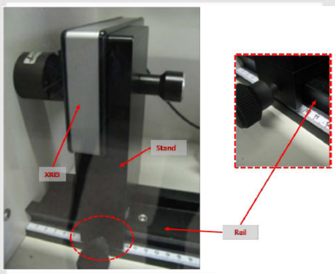

Fig. 5: XRIS setup

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Setup (2/2)

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- 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. 6). 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. 7)
- You can change the High Voltage and current of the X-ray tube in the corresponding input windows or manually on the unit (Fig. 7). When clicking on the unit pictogram additional information

Fig. 6: Commectio of the computer

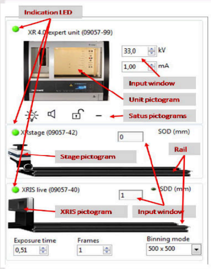

Fig. 7: Part of the user interface of the software

Procedure (1/7)

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1a) Take an offset-Image.

- Start a new experiment, give it a unique name and fill in your details (Fig. 8). Alternatively it is also possible to load this experiment with pre-recorded images. The correct configuration will be loaded automatically as well.
- Adjust the XRIS settings and X-ray unit settings according to Fig. 9 or load the configuration from the predefined CTO file 'Experiment 10' (see Fig. 9).

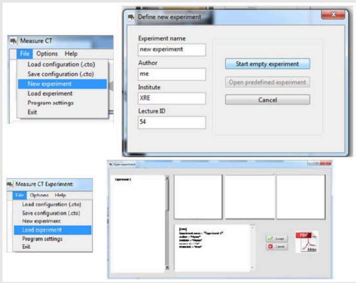

Fig. 8: Start a new experiment and fill in your details

Overview of the settings of the XRIS and X-ray unit:

- 35kV, 1.00mA
- exposure time 20 sec
- Number of frames: 1
- Binning mode 500x500
- SDD= 100

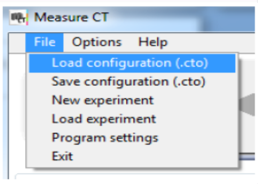

Fig. 9: The settings for this experiment and the method load and adjust the settings

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Procedure (2/7)

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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. 10). 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.
- 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. 11).

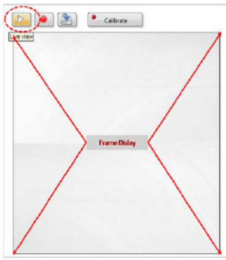

Fig. 10 'Live View' button and blinking green light

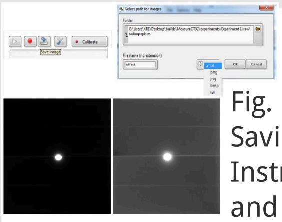

Fig. 11: Saving Instructions and resulting Image

Procedure (3/7)

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1b) Take the Laue diffraction pattern.

Now place the LiF-crystal in the holder (Fig. 12) and move the XRIS to the same position (approx. 9 cm) as in task 1a).

- 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.

Fig. 12: Place the crystal in the holder

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Procedure (4/7)

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1c) Processing of the image.

- To further process an image you need to open the Image Viewer (Fig. 13). 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).
- To process images that you saved in tasks 1 a) and b), open the destination directory (Fig. 14) by choosing "Image" and "Select Image path" (if you started an experiment before performing steps 1 a) and b) the right folder opens automatically).

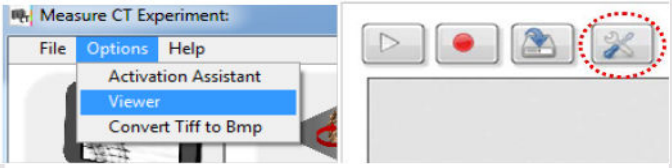

Fig. 13: Open Image Viewer instructions

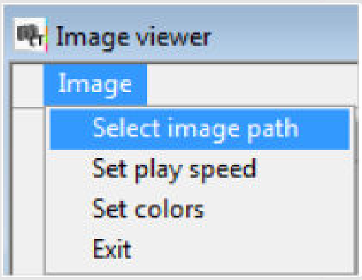

Fig. 14: Select the image path

Procedure (5/7)

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- Double click on 'DI1' from the list, set this image as offset by clicking on "Set as offset" and select the normalisation (Fig. 15). Any new image that you select from the list will from now be subtracted with DI1.
- 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. 16).

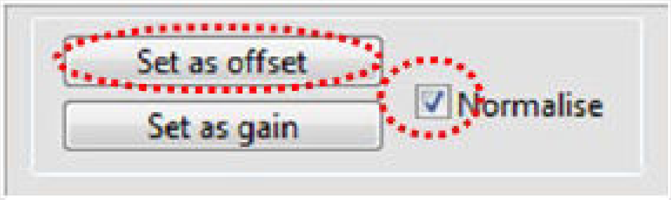

Fig. 15: Correct for offset

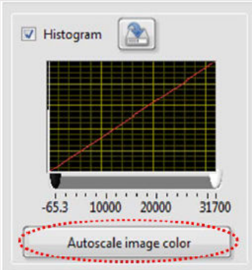

Fig. 15: Autoscale and adjust the Middle colour

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Procedure (6/7)

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- Adjust the upper contrast handls (Fig. 17) 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.

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

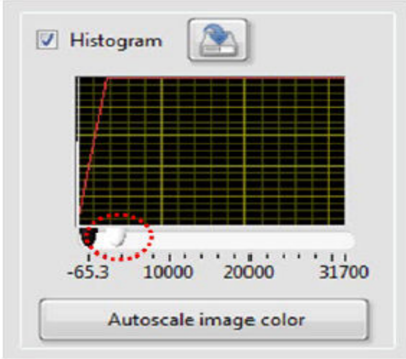

Fig. 17: Adjust the upper contrast handle to the left until the laue patterns become visible

Procedure (7/7)

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- 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. 18) of the displayed histogram by typing in a new upper value (for example 2000).

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

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

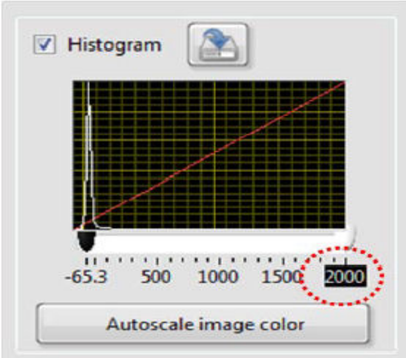

Fig. 18: Adjust the upper contrast level to a smaller value

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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 reflections depends on the reflecting crystal surface as well as on the spectral intensity distribution of the X-rays.

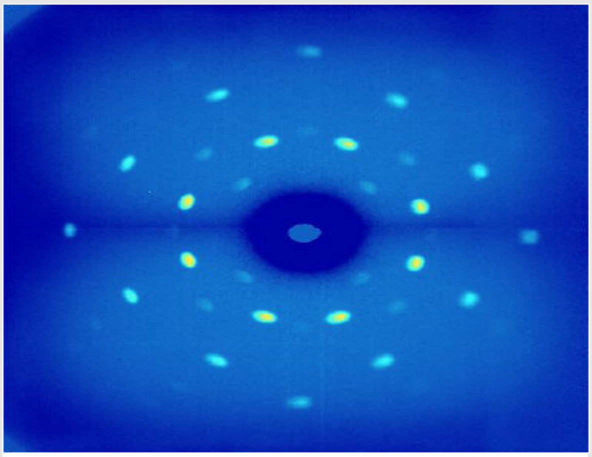

Fig. 19: Laue pattern of the LiF (100) crystal.

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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 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 P2540905), the beginning of the bremsspectrum is given by the minimum wavelength $\lambda_{\min} = 1.24 \cdot 10^{-6} / \mathrm{u_A}\ [\mathrm{m}]$. For an accelerating voltage $\mathrm{u_A}$ = 35 kV, the following is true: $\lambda_{\min}$ = 35.5 pm. This means that for the assignment of the reflections to the lattice planes, only X-rays with a wavelength of \mathrm{\lambda} > 35.5 pm can play a role.

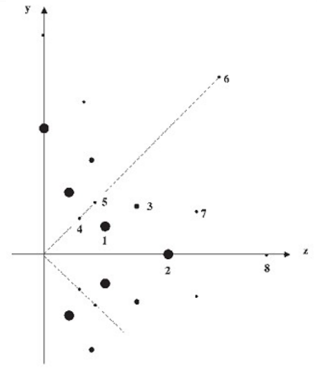

Fig. 20: Schematic representation of the Laue reflections.

Task 2 (part 2)

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 no. 4 and 8 are only very slightly visible in the original photograph. For them a longer exposure time is necessary. 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.

Reflection no.	y [mm]	z [mm]	L [mm]	θ_{exp} [°]	hkl	θ_{cal} [°]	k/l	y/z	d [pm]	λ [pm]
1	4.0	12.5	13.75	17.29	111	17.55	0.33	0.32	121.4	72.2
2	0.0	25.5	25.5	26.66	204	26.57	0	0	100.7	90.4
3	9.75	19.0	21.25	24.17	224	24.09	0.5	0.51	82.2	67.3
4	6.75	6.75	9.5	13.34	133	13.26	1	1	92.4	42.6
5	10.75	10.75	15.5	19.33	244	19.47	1	1	90.1	59.6
6	38.75	38.75	54.5	53.30	111	35.26	1	1	232.6	268.8
7	7.0	34.0	35.5	30.75	315	30.47	0.2	0.2	68.1	69.6
8	0.0	45.75	45.75	33.72	406	33.69	0	0	55.8	62.0

Table 1: Evaluation of the Laue diagram

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Note

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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.

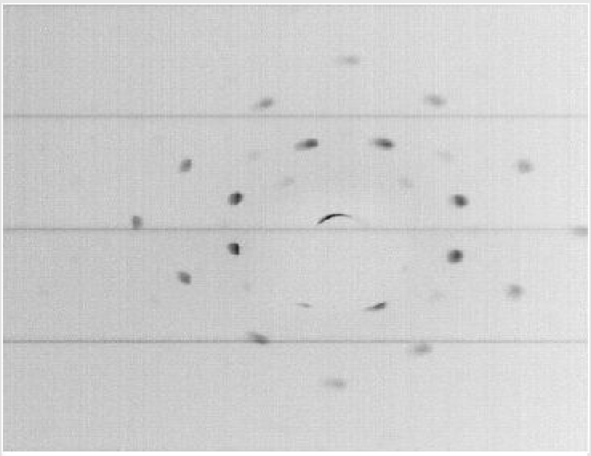

Fig 21: Laue diagram with Beamstop.

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