Department of Physics UZH

Manual for the experiment

Laue X-Ray Diffraction

PHY220 Advanced Laboratory Course in Solid State Physics

September 26, 2023

1   Goal of the experiment

In x-ray crystallography, the Laue method is a simple and fast way of recording a diffraction pattern of a single-crystalline sample. The goal of this experiment is to take diffraction patterns of several samples of *a priori* unknown symmetry using the Laue method. The samples have to be aligned and rotated by means of a goniometer such that a symmetry axis is aligned with the center of the image. From the pictures one may deduce some symmetry properties and thus the basic point group of the crystal lattice.

You can find information about the basic concepts of x-ray diffraction and a discussion of the Bragg or von Laue conditions in almost every solid state physics textbook, like for example:

- N. Ashcroft and N.D. Mermin Solid State Physics, chapter 6 *Determination of Crystal Structures by X-Ray Diffraction*,

- Ch. Kittel, *Introduction into Solid State Physics*, chapter 2 *Wave Diffraction and the Reciprocal Lattice*,

- (in German) K. Kopitzki (and P. Herzog), *Einführung in die Festkörperphysik*, Kapitel 1.2 *Kristalle als natürliche Beugungsgitter*.

Examples for suitable keywords are *Crystal structure*, *diffraction*, *Bragg conditions*, *Laue conditions*, or *Ewald sphere*.

Some key questions which you should be able to answer, and which may be used as guideline during the initial discussion:

1. What are Bravais lattices and point group symmetries?

2. What are conditions for constructive interference for a wave of wavelength λ scattered from a lattice with a layer spacing a?

3. Define the Bragg conditions.

4. Rewrite these conditions for the reciprocal lattice (Laue conditions).

5. What is the Ewald construction?

6. What is the difference between powder diffraction (Debye-Scherrer method), the Laue method, and *rotating crystal diffraction*? How do the diffraction images look like in the three cases?

7. What do the atomic form factor and the structure factor represent? How does the intensity of the scattered x-rays depend on the atomic number Z?

2   Experimental setup
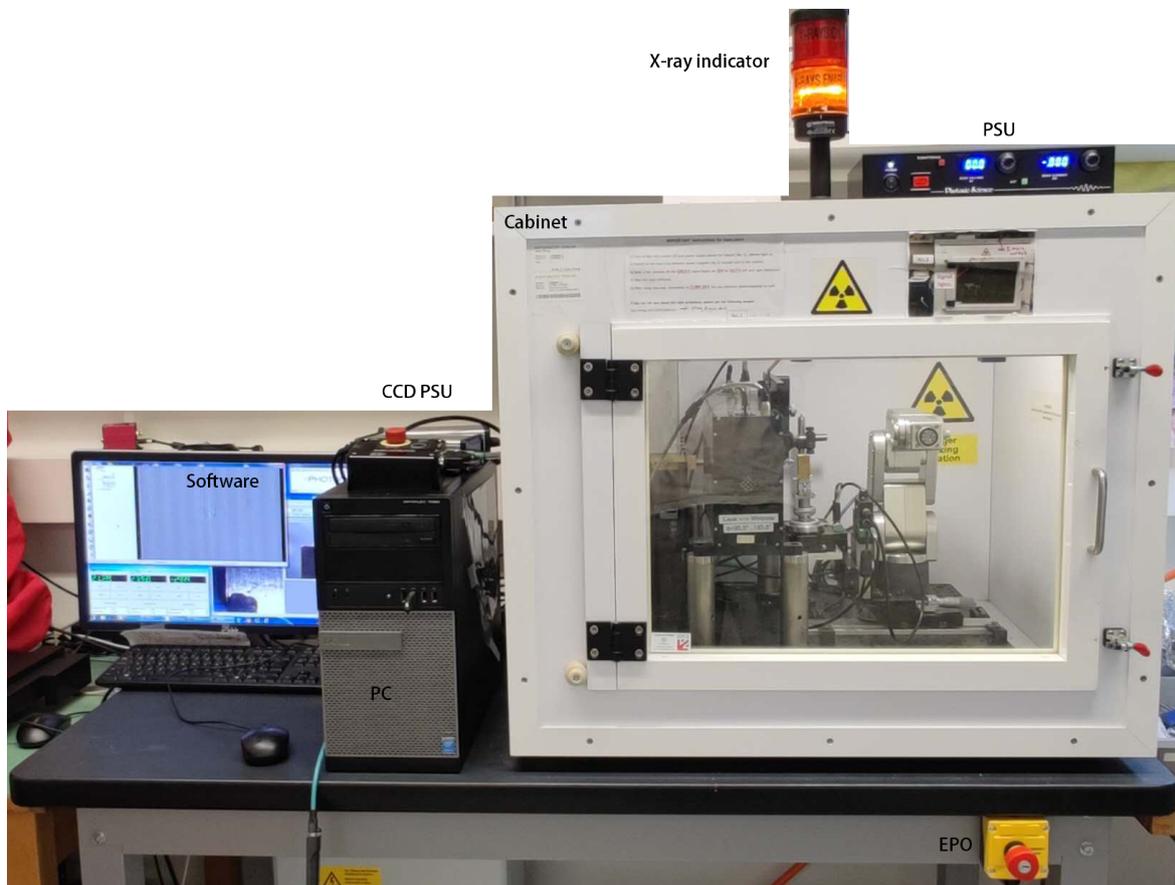
Figure 1: Photograph of the device housing the High-Resolution MiniFocus x-ray source model PXS11-5025 of Thermo Fisher. It is composed of X-ray cabinet with X-ray indicator, a power supply unit (PSU), an emergency power off (EPO), and PSU for CCD camera, as well as data acquisition PC and software. X-ray indicator shows red warning light while the x-ray source is running. The whole cabinet is shielded using lead glass and secured by interlocks against accidental opening of the front door while the x-ray source is running.

A photograph of the experiment is shown in Fig. 1. As shown in Fig. 2, the x-ray exit tube is in the center of the flat detection screen for which only a protective layer is visible from outside. The pattern is thus recorded in backscattering geometry, i.e. in reflection from the sample. The backscattering geometry is necessary because solid samples of the size of typically 1 mm^3 are not transparent to x-rays with photon energies in the range of 10 to 50 keV. Indeed, with the available energy, x-rays can penetrate only a few hundred microns inside the material, and recording the pattern in reflection yields a much higher signal strength than in transmission.

The components of the setup will be described in the following.
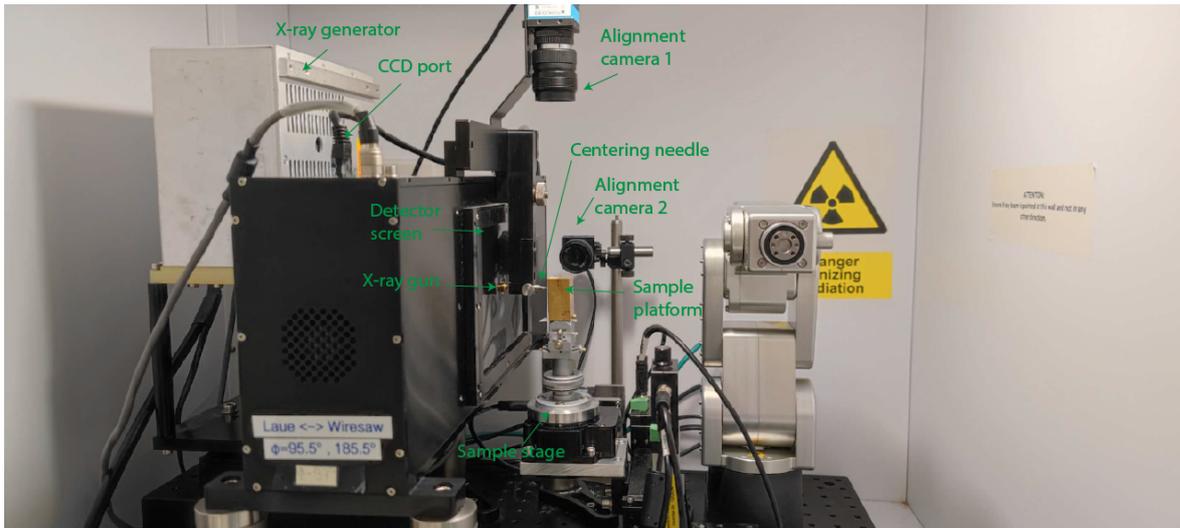
Figure 2: X-ray cabinet. Two CCD cameras are installed behind the detector screen (only the protective surface layer is visible) inside the black box. Center of the detector screen is the exit hole of X-ray gun. The sample is glued onto a sample platform with goniometer (see Figs. 3 and 4) which will be aligned with the x-ray beam assisted by the centering needle which will be turned 90 degree off to clear the light path of X-ray while measuring. Alignment of samples is also assisted by the two alignment cameras. Two translational and one rotational degrees of freedom of the sample stage are motorized and can be controlled by the PC.

2.1   X-ray Source

The X-ray source[1] is a High-Resolution MiniFocus X-Ray Source (model PXS11-5025). It consists of a tungsten target (anode) towards which electrons are accelerated using an acceleration voltage of up to 50 kV. At 50 kV the beam size is 65 μm at the focus position. This sets the highest energy than can be obtained with this source.

The total power of the x-ray source is given by the product of electron current times acceleration voltage which corresponds to the maximum rating of 0.5 mA times 50 kV for the system used here. Only 1% of the electron kinetic energy dissipated in the tungsten anode is re-emitted as radiation, the remaining 99% heat the anode. No water cooling is required because air cooling system is sufficient to dissipate roughly 25 W of electric power converted into heat at the anode.

2.2   Alignment Cameras

The two cameras used for the alignment are CMOS based[2] with an optical zoom lens system.

2.3   X-ray Detector

The x-rays are detected on a large flat screen which is monitored using two cameras.

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[1]A source which produces a collimated beam is often called a "gun".

[2]Two kinds of detectors are often used for image acquisition: Charge-coupled devices (CCD), in which each pixel is made as a potential well accumulating charges, which are read out in series by a charge amplifier. Complementary metal-oxide-semiconductor (CMOS) denotes a technology to produce diodes or transistors. So-called CMOS image sensors have a pn-junction acting as photodiode per pixel. The latter can be read out in parallel. CMOS sensors are usually used as camera sensors in mobile devices.

The detection screen is made out of a scintillating material. When the screen is hit by a high-energy particle or ionizing radiation, like here the diffracted x-rays, the energy is absorbed and re-emitted as visible light which in turn is captured by the cameras. A Gadox (gadolinium oxysulfide, Gd_2O_2S) scintillator doped with Tb (15 mg/cm^2) is used in this system. Gadox is an insulator which, due to the large number of electrons in Ga atoms, has a large absorption cross-section for x-rays. When a diffracted x-ray hits the screen it excites an electron from a core state to a conduction band state. The resulting electron-hole pair forms a stable exciton particle which propagates in the material until a dopant site is reached. The large band gap of Gadox prevents non-radiative recombination of electron and hole, while the additional (in-gap) electronic states at dopant sites provide more channels for radiative recombination. The energy is dissipated by emission of a photon (sometimes together with phonons). This process is called luminescence. The photon energy is given by the energies of the dopant states. In our system, luminescence occurs mostly at the dopant sites, and the luminescence light is emitted in the wavelength range of 382 to 622 nm with an emission maximum at 545 nm (green-yellow). The main advantage of this type of scintillator is the high density of the material which enhances the absorption and, thereby, the sensitivity. Moreover, the spatial "pixel" resolution of the detector is then given by the imaging camera used.

Two cameras are used to acquire the Laue pattern from the scintillator screen. These are two 1280×1960 pixel CCD cameras (see footnote in section 2.2) with a 10 MHz pixel readout rate (note that the total readout time is twice as large as for one camera because they are read serially). The possible exposure times are in the range from 1 ms to 30 min. These two cameras are enclosed in the light-tight box containing the x-ray gun. In this way the image is acquired in a dark environment in order to reduce the noise and thus allow for high-quality data. Two cameras are used in order to avoid the shadow produced by the x-ray gun. One camera is used on each side of the screen and the resulting images are stitched together by the image processing software. For more information, please, refer to the manuals.

2.4   Sample Holder and Manipulator

These components are used in order to align the sample with the beam spot and to control its orientation. With the help of the alignment cameras or else by eye and the centering needle, the sample is aligned with the x-ray gun using the y and z movements (*Zaber* stage, see software section 3). The position of the beam and the optimum sample position along x are indicated by the centering needle which can be mounted on top of the x-ray gun exit hole.

By means of the approaching screw x the distance between the screen and the sample can be changed (see Figs. 2 and 3). The angles (θ on the manipulator and χ and φ on the goniometer, see Fig. 4) are used to find the high-symmetry planes and to orient the sample along a crystal symmetry axis.

3   Software

Important! Before starting any software power on the machine!
The different items and the corresponding programs are listed here. The assistant will explain the use of the software (Fig. 5).

1. **Motors**
   Note that when opening the program, the controller drives the motors to their to initial "home"
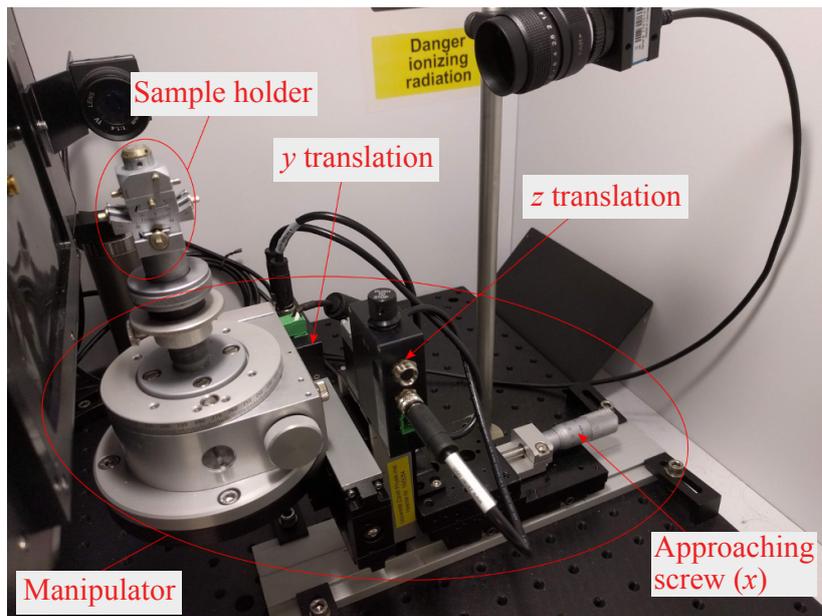
Figure 3: Detailed view of the sample manipulator with the sample holder (goniometer) mounted on three translation stages x ("approaching screw"), y, and z. The latter two are motorized (*Zaber* stage).

position. Be careful that the manipulator and sample holder do not touch the housing or the fragile protection layer of the screen, or else the centering needle if it is in horizontal position!

- Connect the motors to the power outlet.
- Open "PSLViewer"
- Navigate to: Tools $\rightarrow$ Stage control (ZABER)
- Set position to: $z = 5$ and $y = 24$ (values as of 2022 – please ask the assistant for best initial values).
- Reduce the "step size" to 1 or lower for fine adjustments of the position.

2. **Alignment cameras**
   Open "IC Capture". If mounted, the two alignment cameras should already be focused on the right spot, so no further action is required. If needed adjust the camera which is positioned vis-à-vis of screen and x-ray-gun. If the cameras are not used, the alignment can be done by eye using the needle and taking fast x-ray images when the position seems well aligned. The final adjustment should be done by optimizing the image contrast and quality.

3. **X-ray images**
   Turn on the two control stages for the camera: They are underneath the plug for the motors and between the computer and the x-ray diffractometer.

   - Open "PSL Viewer".
   - Navigate to: Camera $\rightarrow$ DualFDS.
   - Click on "Setup" and set the "exposure time" to the desired one (first tests about 5 s, image acquisition several minutes).
   - Click "Snap" for a single shot acquisition or "Play" for a continuous one (recommended to center small samples).
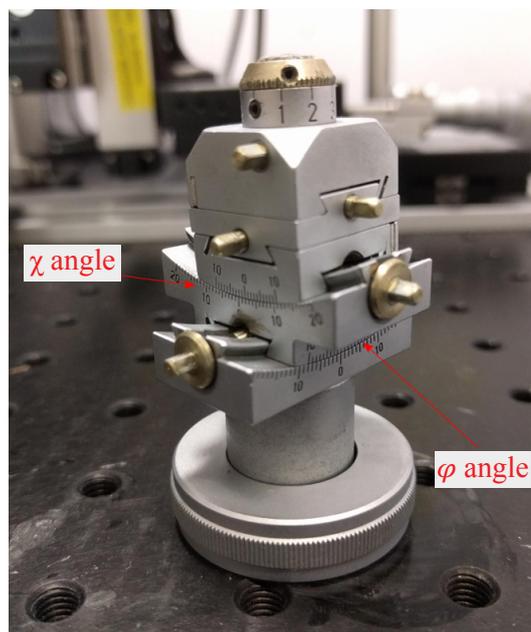
Figure 4: The sample holder (top) mounted on a two-angle precision goniometer (angles χ and φ). Additionally the sample can be moved in two dimensions perpendicular to the center axis in order to align the sample laterally with the pivot point of the goniometer.

4. **Laue simulations**
   Since x-ray diffraction images taken using the Laue method are not easy to interpret, a simulation tool is used for estimating rotation angles needed to orient the samples along high-symmetry directions. The program "QLaue" is used for this purpose and the assistant will explain how to use this.

   Be aware that you will have to set the correct length scale of the image. This is done using two parameters, the distance d between sample and screen (which can be measured using the procedure explained in 4) and the length of an image pixel. For doing the latter navigate to Laue:Set Image:Scale. The physical size of the detector should be given in the software. Note that the number of pixels in your image depends on the binning used. Binning means that several pixels are combined into a new larger pixel, i.e. with "2×2 binning" one has half as many pixels in the two dimensions but with increased signal intensity.

4   Experiment

1. **Align the sample.**
   Observe the sample and try to understand what are the high-symmetry faces from the edges. Using the wax fix the sample #1 on the sample holder, which will be mounted on the sample manipulator. By means of the horizontal and vertical motors align the sample with the x-ray gun. Using the approaching screw, position the sample in the focus of the beam. Turn on the instrument and check the alignment by acquiring one image (tip: set exposure to 10-15 s). If no signal is detected change the horizontal and/or vertical position. Once the sample is found, determine the optimal exposure and acquisition mode.

2. **Find the high-symmetry planes and determine the symmetry elements.**

Figure 5: Data acquisition software (PSViewer) and alignment camera (IC Capture). Sample alignment is assisted by two motorized translational and one rotational degrees of freedom of the sample stage which is controlled under PSViewer/Tools/Zaber. Data acquisition is accessed under PSViewer/Camera/DualFDS, image can be exported under PSViewer/File.

Using the θ angle (and, if needed, χ and ϕ) rotate the sample until a high symmetry plane is found. Save the image. Continue to rotate θ and acquire images until about 90 degrees are reached with respect to the initial position. Using the acquired images, determine the crystal symmetry of the sample.

3. Repeat tasks 1 and 2 for another sample #2.

4. **Get scaling parameters for the simulation.**
   Measure the distance of the sample to the detector screen. The latter is about 5 mm behind the protection surface and shall be determined experimentally using a sample which is aligned and oriented with a high-symmetry axis in its center:

   - Write down the *x* position of the sample.

   - Take an image and identify principal diffraction spots. Measure their distance to the center of the diffraction pattern.

   - Move the sample along *x* by a defined distance, i.e. 20 mm.

   - Take a second image: find the same diffraction spots (in which direction should they move?) and determine their distance to the center of the pattern.

   - Use the proportionality theorem (German: *Strahlensatz*) to determine the precise distance between sample and detector screen.

5. **High-symmetry planes and comparison with simulation.**
   Align again the two samples on the [001] and [100]/[010] low-index faces and acquire high-quality data. Use all the possible rotating angles to align the samples with high precision on the

center and horizontally/vertically. Check that these planes are correct by using the simulation tool.

Some examples of the space group and lattice parameters of crystals, which you might investigate, are given in the table below.

Sample	a [Å][1]	b [Å]	b [Å]	α [deg][1]	β [deg]	γ [deg]	Space group
Si	5.42	5.42	5.42	90°	90°	90°	Fd$\bar{3}$m (227) fcc diamond
Cu	3.615	3.615	3.615	90°	90°	90°	Fm3m (225) fcc
Sr$_2$RuO$_4$	3.87	3.87	12.75	90°	90°	90°	I4/mmm (139) tetragonal
IrTe$_2$	3.93	3.93	5.38	60°	120°	120°	P3m1 (164) trig. (1T polymorph)

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[1]In this table, the lattice parameters refer to the so-called conventional unit cell: the crystal axes are chosen along high-symmetry directions. This is different to the primitive cell. E.g., silicon crystallizes in the diamond structure which contains two fcc lattices shifted by $\left(\frac{a}{4} \frac{a}{4} \frac{a}{4}\right)$ with respect to each other. The Si-Si distance is then $\sqrt{3}\frac{a}{4} = 2.35$ Å.

5   Questions to answer in your report

1. How are x-rays generated?

2. What symmetries do you observe in the recorded patterns?

3. Deduce the crystal orientation from the symmetries and the known symmetries of the Bravais lattice.

4. How would the patterns change in case of a monochromatic x-ray source like e.g. a synchrotron light source?