LAUE
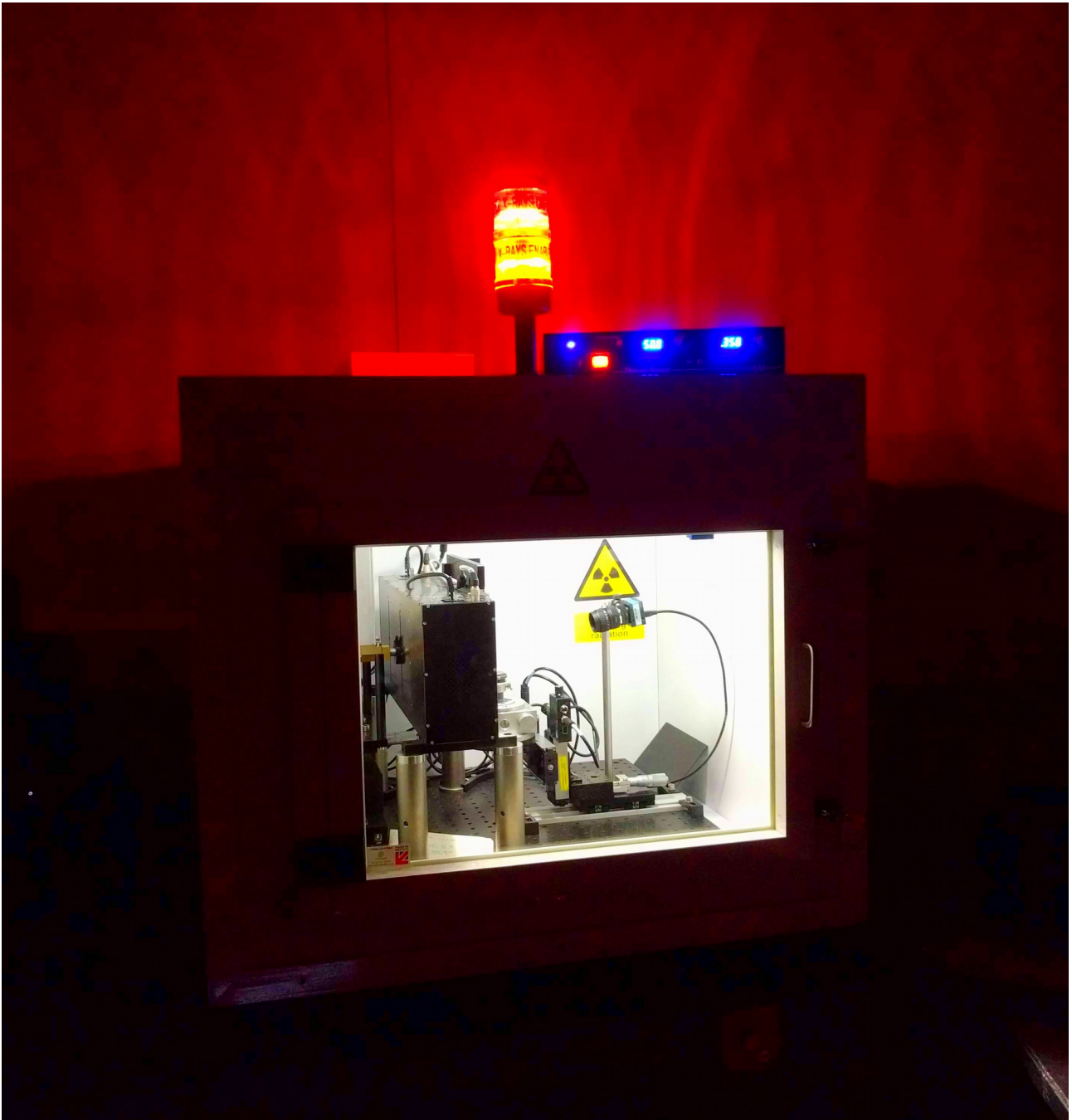
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Summary

1 – <u>Instrument</u>:
- X-ray Source
- Cameras
- Scintillator (Screen)
- Manipulator & Sample Holder

2 – <u>Software</u>:
- Motors
- Cameras
- Laue

3 – <u>Experiment</u>:
- Align the samples
- Find the high-symmetry planes and determine samples' symmetry
- Compare with simulation

Instrument

The instruments adopts a backscattering geometry. Thus, as illustrated in Figure 1, the X-ray gun is in the middle of the detection screen (for which just a protective layer is visible). The backscattering geometry is necessary due to the "soft" nature of X-rays. Indeed, with the available energy, x-rays can penetrate only a few hundred of microns inside the material but a usual size of the samples is about 1 mm^3.
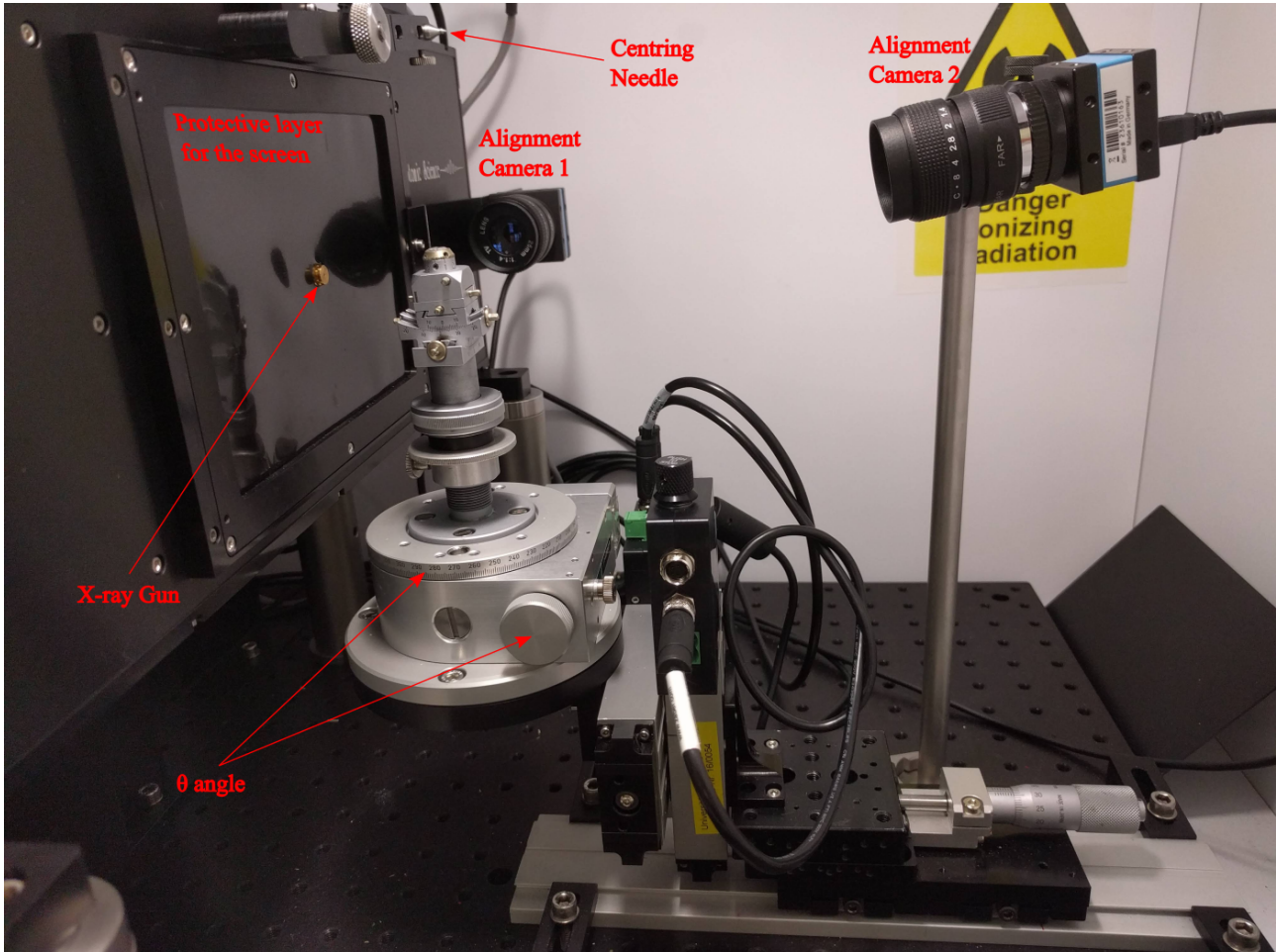

Figure 1

1 – X-ray Source

The X-ray source is a High-Resolution MiniFocus X-Ray Source (model PXS11-5025). It consists of a Tungsten target on which electrons can be accelerated with a maximum voltage of 50kV. At 50kV the beam size (at the focus position) is 65 μm. This sets the highest energy than can be obtained with this source.

No water cooling is necessary since an air cooling system is sufficient.

For more information see the data sheet.

2 – Cameras

The two cameras used for the alignment are CMOS based with optical zoom system.

Other two cameras are used to acquire the Laue pattern on the screen. These are two 1280 x 1960 pixel CCD cameras with 10 MHz pixel rate (readout time is as twice as bigger because they are read serially). The possible exposure times are in the range from 1 ms to 30 minutes.

These two cameras are enclosed in the 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 allowing to obtain high-quality data.

Two cameras are used in order to remove the shade from the x-ray gun. Thus, one camera is used on each side of the screen and the resulting images are synchronised and processed subsequently. For more information refer to the manuals.
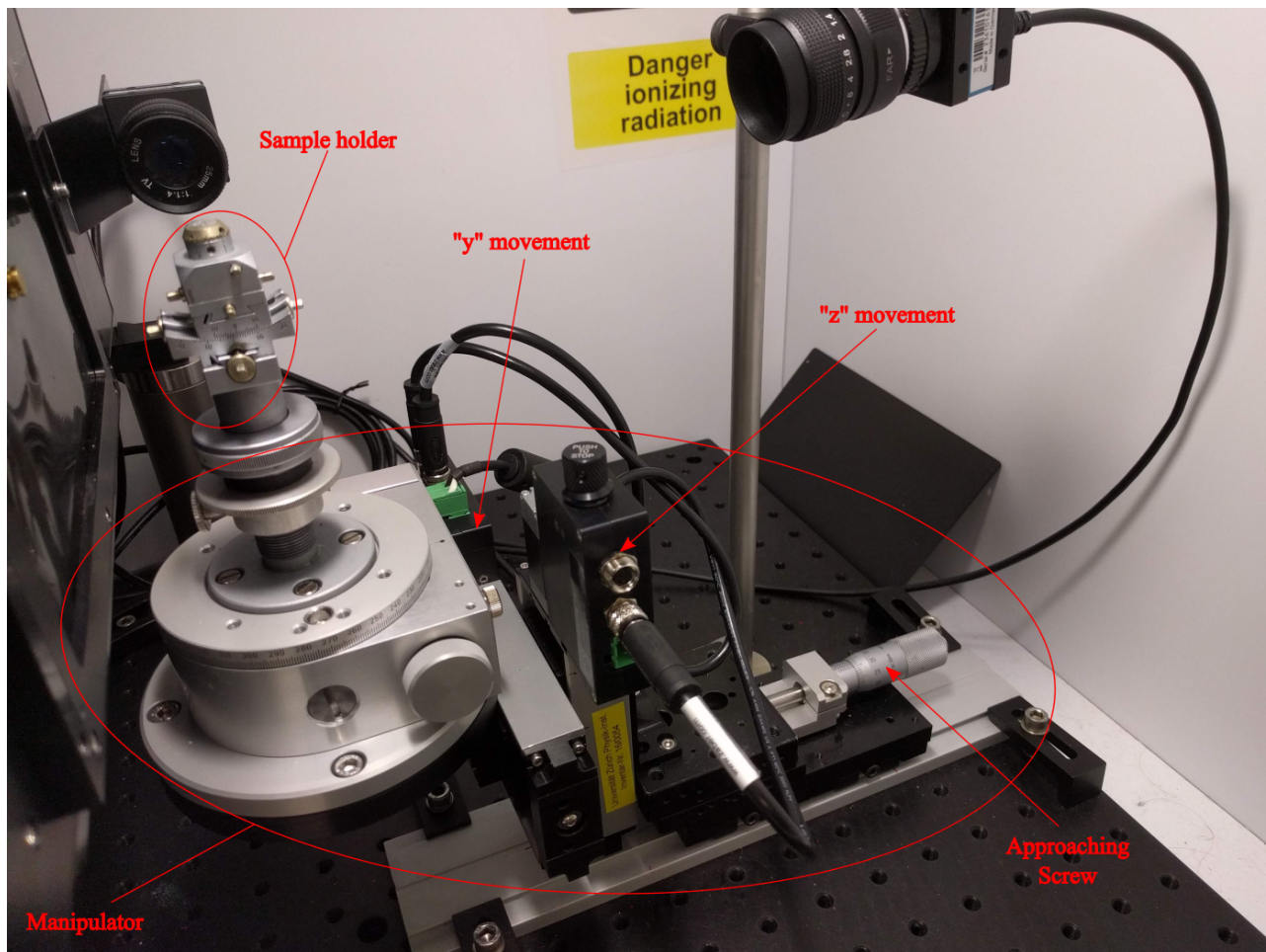

Figure 2

3 – Scintillator (Screen)

The detection screen is composed of a scintillating material. When the screen is hit by a ionizing radiation (i.e. the diffracted x-ray signal) it absorbs it and re-emits in visible light (which in turn is captured by the CCD cameras).

A Gadox (Gd_2O_2S) scintillator doped with Tb (15 mg/cm2) is used in this system. Thus it emits in the range of 382 - 622 nm with a primary emission at 545 nm. Note that this particular type of scintillator is used because it is a high density material. Nevertheless this does not set the "pixel" resolution of the screen but enhances the sensitivity. Indeed, when a diffracted x-ray hits the screen it creates a core-hole (exciton) particle which propagates in the material until a dopant site is reached. Thus a luminescence occurs at the dopant sites only. This is because Gadox is an insulator and thus presents a band gap. On the other side, Tb, being a dopant, creates states inside this gap. Thus when an exciton arrives at a dopant site a recombination mechanism is given and a luminescence occurs.
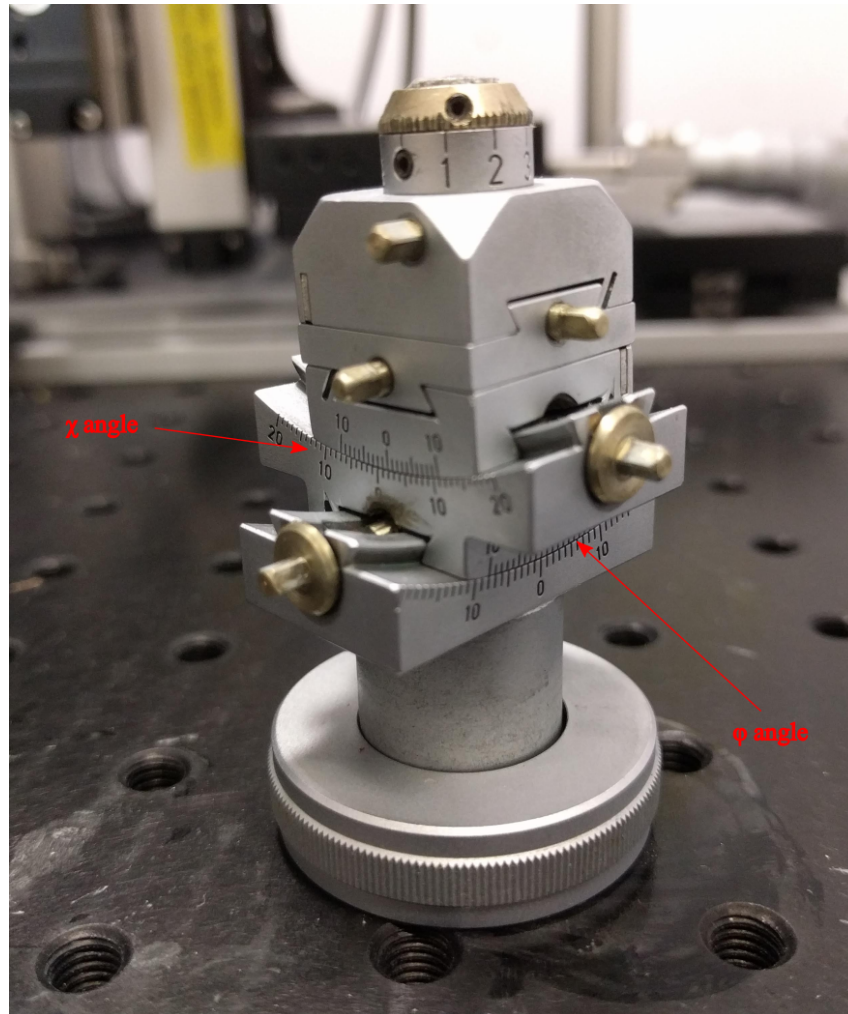
Figure 3

4 – Manipulator & Sample Holder

These components are used in order to align the sample with the beam spot and to manipulate its orientation. With the help of the alignment cameras the sample is aligned with the x-ray gun using the y and z movements, and by means of the approaching screw, positioned on the beam focus (see Figures 1 & 2). The later is indicated with the centring needle when it is in vertical position.

The angles (θ on the manipulator and χ & φ on the sample holder) are used to find the high-symmetry planes.

Software

Before opening any software turn on the machine!

1 – Motors

Notice! When open drives the motors to their to initial position. Be careful that the centring needle is in horizontal position and that the manipulator and sample holder do not hit the screen box.

Connect the motors to the power outlet.
Open "PSLViewer"
Navigate to: Tools → Stage control (ZABER)
Go to: 10 in z and 12.5 in y
Reduce the "step size" to 1 (or lower) to fine tune the alignment

2 – Cameras

        Open "Cameras.icf"

        The two alignment cameras are already focused on the right spot, so no further action is required. If needed adjust the camera with looks directly on the x-ray-gun.

3 – Laue

        Turn on the two control stages for the camera! These are under the plug for the motors and between the PC and Laue.

        Open "PSL Viewer"

        Navigate to: Camera → DualFDS

        Click on "Setup" and set the "exposure time" to the desired one.

        Press "Snap" for a single shot acquisition or "Play" for a continuous one (recommended to centre small samples)

Experiment

1 – Align the sample

        Observe the sample and try to understand what are the high-symmetry faces from the detects/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/vertical position.

        Once the sample is found, determine the optimal exposure and acquisition mode.

2 – Find the high-symmetry planes and determine samples' symmetry

        Using the θ angle (and eventually χ and φ) rotate the sample until high symmetry plane is found. Save the image.

        Continue to rotate θ and acquire images until about 90-100 degrees, from the initial position, are reached.

        By the acquired images, determine the crystal symmetry of the sample.

Repeat point 1 and 2 for samples #2a and #2b.

3 – Compare with simulation

        Align again the two samples on the [001] and [100]/[010] faces and acquire high-quality data. Use all the possible rotating angles to align the samples with high precision on the centre and horizontally/vertically. Check the these planes are correct by using the simulation tool. The space group and lattice parameters of the samples are given below.

Sample	Composition	a [Å]	b [Å]	c [Å]	α	β	γ	Space group
#1	Sr_2RuO_4	3.87	3.87	12.75	90	90	90	139 (I4/mmm)
#2	$IrTe_2$	3.93	3.93	5.38	60	120	120	164 (P3m1)