University of Regensburg

Advanced Physics Laboratories

Experiment:
X-Ray Diffraction

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Contents

1. Warning and safety instructions

X-rays, especially the "soft" radiation from a Cu anode, are strongly absorbed in the human body and can cause burns and incurable damage to affected tissue parts in fractions of a second. The radiation is invisible as long as it is not made visible, e.g. by fluorescent screens. Therefore, special caution is required when handling X-ray sources. For this reason, we ask you to read the excerpt from the X-ray ordinance available in the lab course and to absolutely follow it [1].

2. Basics and preparatory questions

By the time of the preliminary discussion, you should be familiar with the following basics, most of which can be found in [2, 3]:

(a) X-ray radiation:
Generation, detection and use of X-rays; In the first part of the experiment, a camera is used in which a phosphor screen is coupled to a CCD array (see Appendix B). Therefore, also inform yourself about the operating principles of a CCD array [4].

(b) Crystallography:
The concept of a crystal (especially translational lattice, crystal systems and Bravais lattice) and its symmetry as well as the description of simple crystal structures such as those of NaCl or KCl, CsCl and Si. You should also learn about Miller's indices and the denotation of lattice planes in crystals. In addition, an important concept is that of the reciprocal lattice. What relationship can be derived from this for the spacing of lattice planes d_{hkl}?

(c) X-ray diffraction on crystals:
Bragg and Laue equations, Ewald construction, basic concepts of crystal structure determination.

In addition, you should already learn about the evaluation procedures for the Debye-Scherrer procedure and about the indexing of Laue images (see section 4). What is a Nelson-Riley plot [6, 5]?

As you work through item (c), also familiarize yourself with the following issues:

(d) What do the extinction rules for X-ray diffraction on crystals with fcc structure say? More generally formulated: What determines the intensity of a Bragg reflex? I.e. why are some reflections only very weak or not present at all? Terms like lattice factor, structure factor and atomic shape factor help here.

3. Experimental setup

The following devices are present at the workplace, as can be seen in Figure 1. (This is technology from four decades!):
A powder diffractometer (a) with the corresponding measuring computer (b), as well as a diffractometer (c) for the Laue recordings and a notebook (d) for the evaluation of the data.

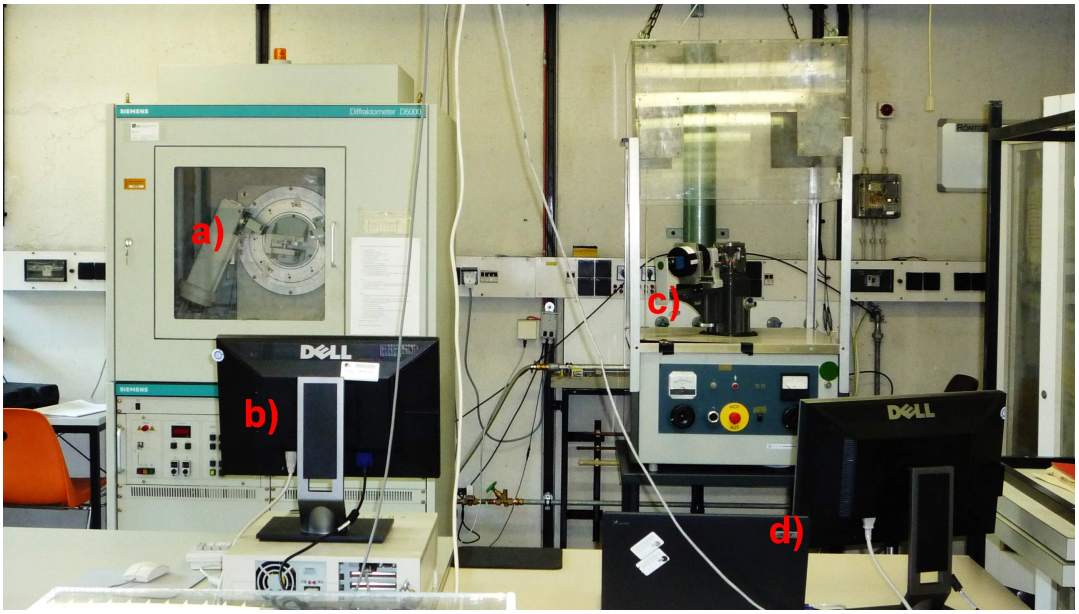


Figure 1: (a) A comparatively new powder diffractometer: In this instrument, a Cu X-ray tube with Ni filter is used to produce monochromatic Cu K_α radiation. The X-ray beam is guided to the sample in Bragg geometry and collected by a scintillation detector. (b) A measurement computer controlling the powder diffractometer. (c) An older diffractometer with a W X-ray tube and a filter drum on the tube cover for the Laue experiments. The diffraction pattern is recorded using a special X-ray camera (state-of-the-art). (d) A Notebook, which is used on the one hand for the control of the X-ray camera of the Laue experiment, on the other hand also for the conversion of the Debye-Scherrer data into ascii files.

OpenOffice 3.3 is installed on the notebook for data evaluation. In addition, QTI-Plot, a program for visualizing your data, is available. A USB stick is required to take your data home. Please collect all your data in a separate folder on the desktop of the notebook.

4. Experimental execution and evaluation

4.1. Laue images

The aim of this part of the experiment is to record diffraction images in Laue configuration of different crystals. The Laue symmetries of NaCl or KCl along the [001] crystal axis, as well as those of Si(001) and Si(111) wafers along the respective growth direction shall be determined. For NaCl or KCl, the Miller indices of the diffraction reflections are also to be identified.

4.1.1. About taking an image and its processing

Attach the X-ray camera to the optical rail so that the base of the camera is aligned with the black markings (see figure 2). The crystal-image plane distance is then 22.1 mm. The × 45 mm field of view of the camera's phosphor screen is guided through a taper onto the × 2048 pixel CCD array (see Appendix B). With this the pixel coordinates can be converted later into diffraction angles.

For an exposure, first of all the water cooling of the diffractometer must be opened and the protection box must be closed. Then slowly increase the voltage to 40 kV and then the current to 30 mA. By setting the filter drum to the "O" position, the "white" X-rays required for Laue diffraction on single crystals are obtained. Finally, the aperture must be opened with the $\boxed{1}$ key so that the X-ray beam exits the aperture.

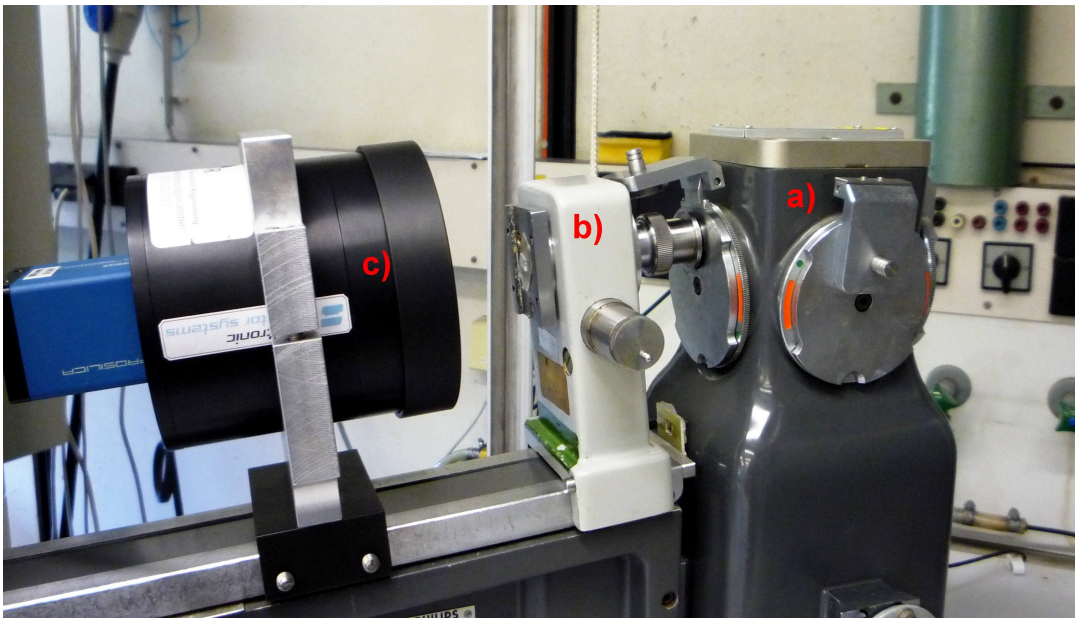


Figure 2: Experimental setup for diffraction on single crystals: (a) X-ray cover with filter drum, (b) crystal holder, and (c) X-ray camera.

All further steps are done with the program "AVT UniCamViewer"' on the notebook. Proceed as described in Appendix A. Do not forget the camera initialization (see also

appendix A). Optimize the exposure parameters to obtain an image with as little noise as possible. Pay particular attention to the following points:

- By "binning", pixels are combined in horizontal and vertical direction to increase the light output per pixel. This reduces the resolution of the array and of course increases the effective pixel size. It is recommended to do a "binning"' of 4×4 pixels to one, resulting in an image size of 512×512 pixels.
- The "gain"' of the CCD array works like an ISO value in common cameras: Here intensity is exchanged for increased noise.
- Exposure times above about 30-45 seconds are not very reasonable, because the CCD array has no cooling and therefore the noise increases strongly with the exposure time (hotpixels).

Despite all carefulness, a clear noise will be visible in the image, since the camera is operated at its detection limit. Therefore, it is recommended to take a dark image with the optimized parameters and to subtract this noise from the actual image. For this you can use e.g. the freeware GIMP installed on your notebook: Image and dark image are loaded in two layers and overlaid by means of "Modus: Abziehen"'. Pay attention to the order of the layers. Once you have united the layers, you can still optimize the brightness values using the dialog "Farben $\rightarrow$ Kurven"'.

You should now have obtained a symmetrical diffraction pattern. If the pattern is strongly distorted, the single crystal must be readjusted. If, on the other hand, it is only slightly distorted, then it is usually sufficient to average the coordinates of equivalent reflections.

4.1.2. Image acquisition and evaluation

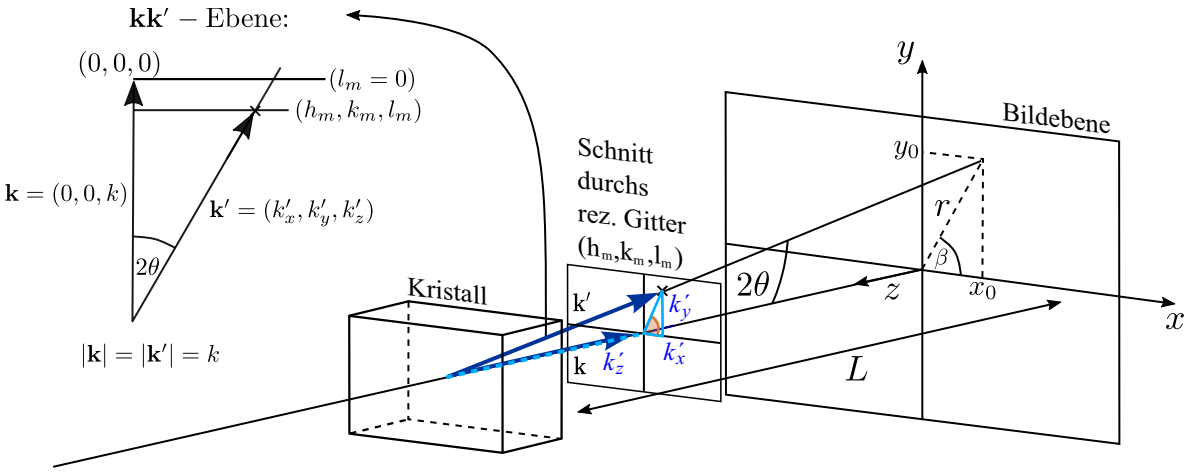
(a) **NaCl and KCl:**
In this part of the experiment, $0.5 - 1$ mm thick NaCl and KCl crystal platelets, which were refracted in the [001] direction, are examined. For this purpose, these platelets are oriented on the crystal holder perpendicular to the primary beam. The following parameters are used for the X-ray source: Voltage 40 kV, current 30 mA, distance 22.1 mm. Determine the symmetry elements of the two images and plot them. Also determine the (hkl) values of the diffraction reflections.

The indexing and discussion of the images of NaCl and KCl should be done with the help of the concept of the reciprocal lattice. For this purpose, derive with the help of the drawing 3 and the Laue equation for cubic crystals in vectorial form

$$\Delta\vec{k} = \vec{k'} - \vec{k} = \vec{G} = \frac{2\pi}{a}(h_m, k_m, l_m) \tag{4.1}$$

the following relationship between the Miller indices h_m, k_m, l_m and the distances on the Laue image:

$$h_m \ : \ k_m \ : \ l_m = x_0 \ : \ y_0 : \ r\frac{1-\cos(2\Theta)}{\sin(2\Theta)} \tag{4.2}$$



Laue-Gleichung: $\Delta\mathbf{k} = \mathbf{k}' - \mathbf{k} = (k'_x, k'_y, k'_z - k) = \mathbf{G} = h_m\mathbf{b}_1 + k_m\mathbf{b}_2 + l_m\mathbf{b}_3 = (2\pi/a)(h_m, k_m, l_m)$

Figure 3: Sketch to derive the relationship between h_m, k_m and l_m and the measured quantities x_0, y_0 and L

A Laue image can thus be understood as a projection of the reciprocal lattice.

Is the indexing unambiguous? And what are the possibilities to verify it? Describe and explain the symmetry elements in the image and draw them in the image. Calculate the effective crystal-camera distance $L_{\text{eff}} = \frac{h}{k}\frac{y_0}{x_0}L$ and correct your results if necessary.

(b) **Si(001)-Wafer:**
Create a Laue image with the parameters given in (a). Again, describe the symmetry elements present and draw them.

(c) **Si(111)-Wafer:**
Again, create a Laue image using the parameters from part (a). In this wafer, the (111) crystal planes are slightly tilted with respect to the wafer surface. Again, describe and explain the symmetry present in the image. Now consider in which direction to the surface the (111) planes lie, and justify your guess. You can reduce the distance between the crystal and the camera slightly to better see the symmetry of the image and its distortion due to the misorientation. Finally, correct the position of the crystal with some wax to create a symmetrical image! Draw the symmetry elements in the image.

4.2. Powder spectra

In this part of the experiment, which is performed on the powder diffractometer with Cu-K_α radiation, the lattice constants of NaCl and KCl are to be determined. The experimental setup is shown in Figure 4.

For this, put optimally crushed material of the crystals into the designated holder of the Θ-2Θ-goniometer [6]. In a standard scan of the sample, the sample now moves one

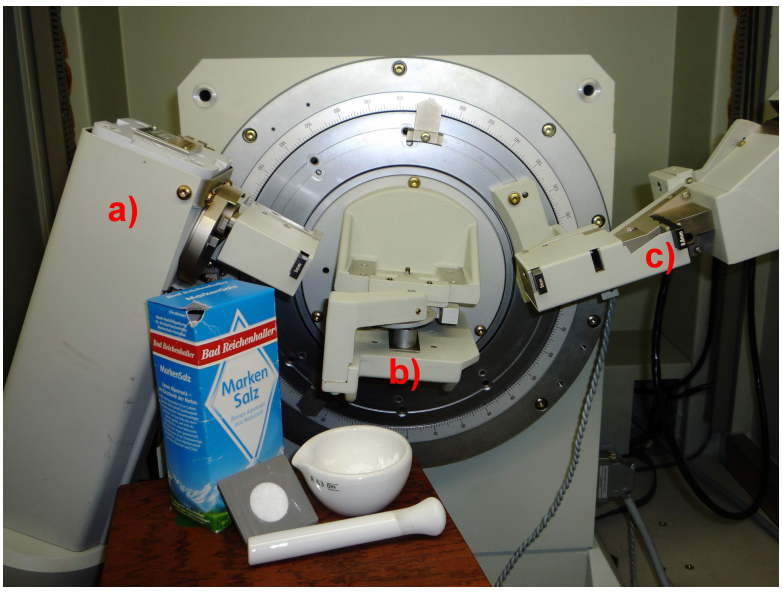


Figure 4: Experimental setup for diffraction on crystal powders: (a) X-ray tube, (b) sample plate, (c) detector. In the foreground a NaCl sample can be seen.

Θ step at a time, but the detector moves one 2Θ step. Thus, a spectrum is obtained which corresponds to a narrow strip from a Debye-Scherrer image.

Spectra from $2\Theta = 5°$ to $135°$ should now be made, each with a step size of $0.1°$. If only a small amount of crystal powder is available, it is recommended to measure a reference spectrum of the holder without powder to identify any reflections from impurities.

The spectra of NaCl and KCl measured by you are available on the measuring computer in the directory C:\USERDATA\PRAKTIKU and can be reformatted after data transfer with diskette on the notebook with the help of the program WinFit into ASCII files. When evaluating the spectra, the procedure for Debye-Scherrer recordings can be applied. Therefore, derive the following equation based on the squared Bragg equation and the explicit insertion of the net plane spacing d_{hkl} in cubic crystals:

$$\frac{4a^2}{h^2 + k^2 + l^2} \sin^2 \Theta = \lambda^2 \tag{4.3}$$

More details can be found in [7, 6, 8]. It is not sufficient to give the lattice constant as an average value from the diffraction angles of the reflections after their indexing via the "quadratic form"' of the Bragg equation. Here, a suitable extrapolation procedure should be applied to detect and account for systematic errors, the so-called Nelson-Riley plot (see [5, Vol. II, p. 218] or [6]).

Discuss the possible errors. How accurately could the lattice constant be determined under optimal conditions? Discuss the observed line widths, especially with respect to the size of the crystallites. What is the origin of the splitting of the lines at large diffraction angles?

References

[1] Bundesgesetzblatt Z 1997A v. 1.3.1973: *Verordnung über den Schutz vor Schäden durch Röntgenstrahlen* (Röntgenverordnung RöV).

[2] C. Kittel: *Einführung in die Festkörperphysik*, Oldenburg-Verlag (2006), Kapitel 1 & 2.

[3] W. Demtröder: *Experimentalphysik 3: Atome, Moleküle und Festkörper*, Springer-Verlag (2006), Kapitel 11.

[4] http://en.wikipedia.org/wiki/Charge-coupled_device

[5] J. S. Kasper, K. Lonsdale (Editors): *International Tables for X-Ray Crystallography Vol. I - IV*, The Kynoch Press (1972).
https://archive.org/details/InternationalTablesForX-rayCrystallographyVol2

[6] L. Spieß, G. Teichert, R. Schwarzer, H. Behnken, C. Genzel: *Moderne Röntgenbeugung, Röntgendiffraktometrie für Materialwissenschaftler, Physiker und Chemiker*, Vieweg-Teubner (2009).

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[8] H. Krischner: *Einführung in die Röntgenfeinstrukturanalyse*, Vieweg Braunschweig (1974).

[9] J. L. Amoros, M. J. Buerger, M. Canut de Amoros: *The Laue Method*, Academic Press (1975).

[10] E. Preuss, B. Krahl-Urban, R. Butz: *Laue Atlas*, Bertelsmann Universitätsverlag (1974).

A. HR75 Interface

The HR75 X-Ray camera is a high resolution CCD camera with a 75 : 25 mm coupling to a phosphor screen through a taper consisting of glass fibers. The phosphor coating is required for sensitivity to X-rays.

A.0.1. Control of the camera:

The camera is delivered together with a notebook. All necessary drivers for the camera are pre-installed. Two programs are available to operate the camera and take pictures. Desktop shortcuts exist for both programs.

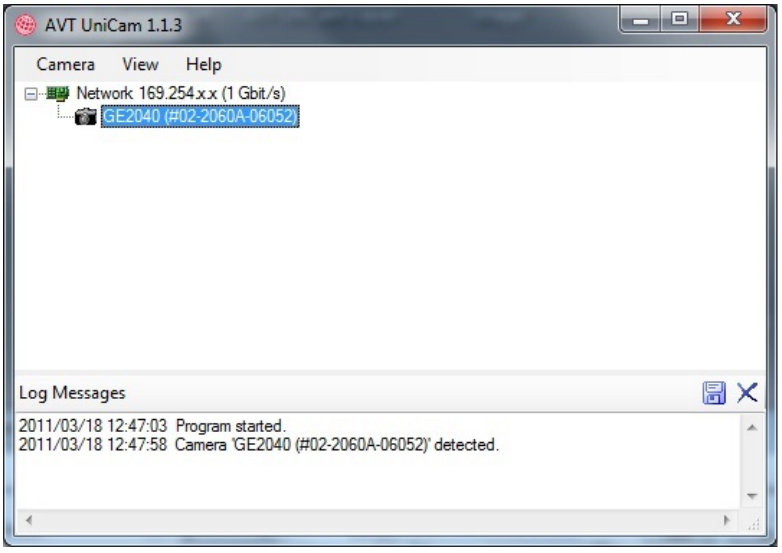
- AVT UniCam Viewer
- ImageJ (with an AVT Plugin), not supported by Proxitronic

The AVT ImageJ plugin is started by the following ImageJ menu path: File → Import → AVT Camera

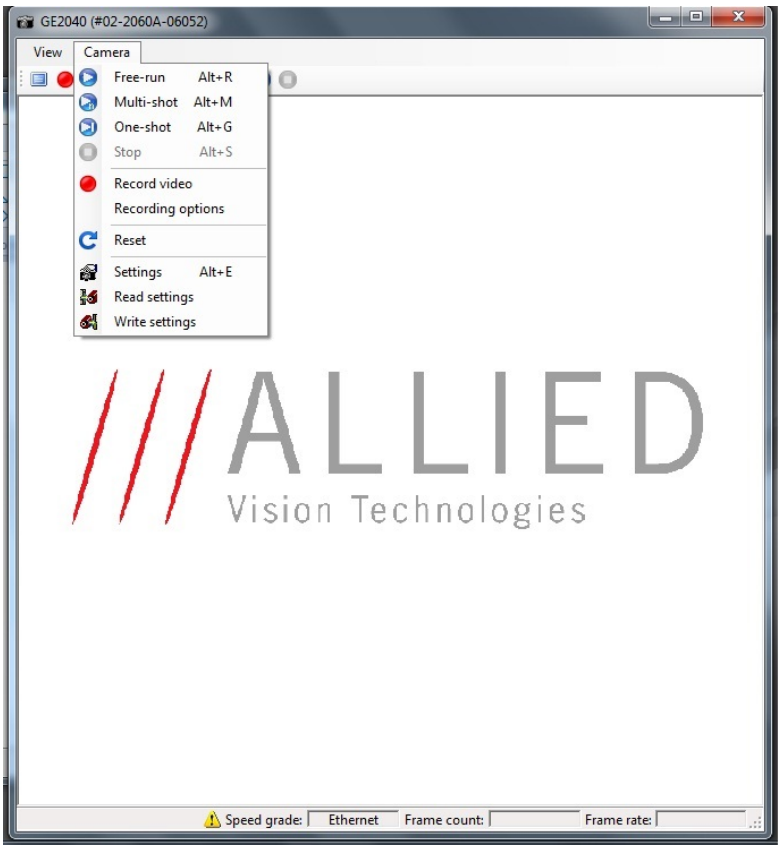
Before starting the plugin, it is necessary to start image capture with AVT UniCam viewer at least once after turning on the computer. (Close the AVT UniCam viewer before starting the AVT plugin).

A.0.2. AVT UniCam Viewer:

Supply the camera with power and connect it to the notebook through a network cable. Then start AVT UniCam Viewer by double-clicking on the desktop icon. It may take up to half a minute until the camera appears in the menu.

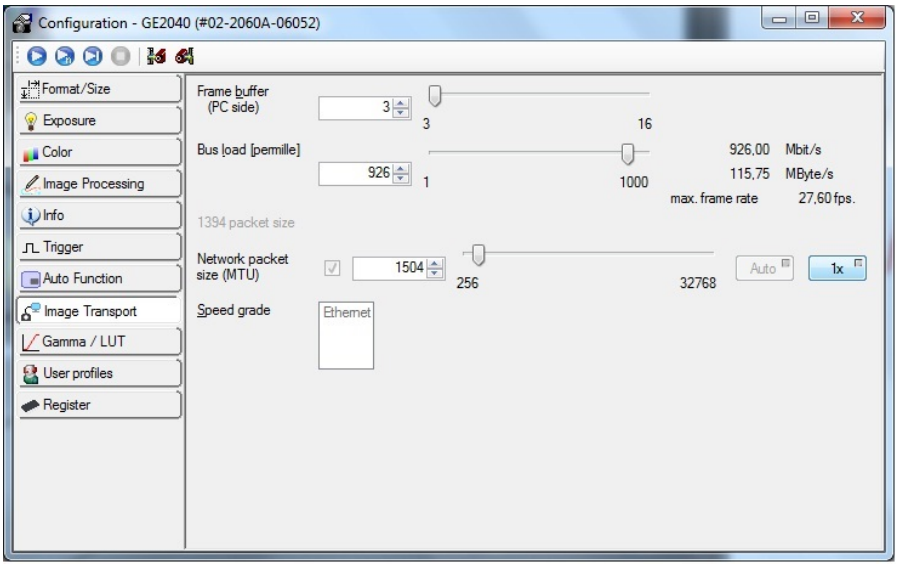


The camera menu is started by double-clicking.

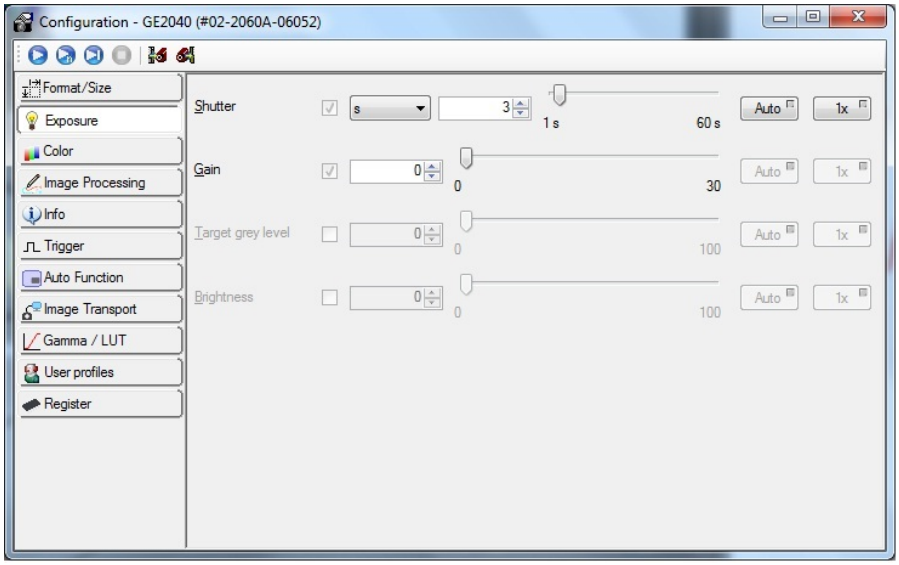


A.0.3. Camera initialization:

Sometimes, especially when the AVT UniCam Viewer is started for the first time or when the MTU (minimal transfer unit) size has been changed, it may be necessary to press the "1x"' button in the "Image Transport"' menu. This initializes the camera.



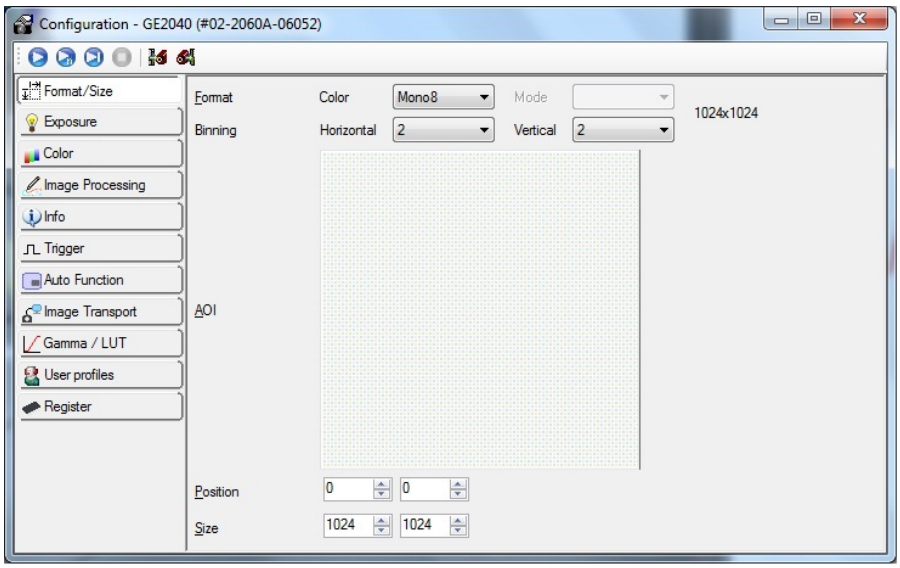
A.0.4. Exposure times:



- In manual shutter mode, relatively long exposure times of up to 60 seconds are possible.
- In auto shutter mode, exposure times are limited to 2 fps (500 ms).

A.0.5. Binning:

To increase the sensitivity of the camera, several pixels of the CCD array can be combined in both spatial directions (binning).



B. HR75 Specifications

Technical data HR75 X-Ray

Camera Specification	
Camera interface	GigE
Framerate	15 fps @ full resolution
CCD sensor	Kodak KAI 4021, 1.2"
CCD pixel	2048 x 2048 (H x V)
CCD pixel size	7.4 µm x 7.4 µm (H x V)
Digitisation	8 bits / 12 bits
Synchronization	External Trigger, fixed frame rate, software trigger
Exposure Control	75µs to 60s
Driver software	Included, ROI, Binning
Camera Specification	
Field of view	45 mm x 45 mm
Phosphor Coating	P43, other on request
Typical sensitivity range	about 20 to 100 keV (x-ray, other on request)
Input window	Aluminium 0,5 mm or different material on customer request (also "open face" for UV or electron sensitivity)
Resolution	Standard type: ≤50 µm, 11 lp/mm (@ 20% contrast) resolution-optimised version available on request

Description

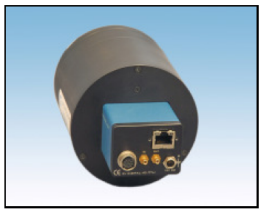
The camera ***HR75 X-Ray*** is a high resolution CCD camera with a 75:25 mm fiber optic taper coupling.

X-ray, UV and electron sensitivity is achieved by phosphor coating (typically P43 phosphor)

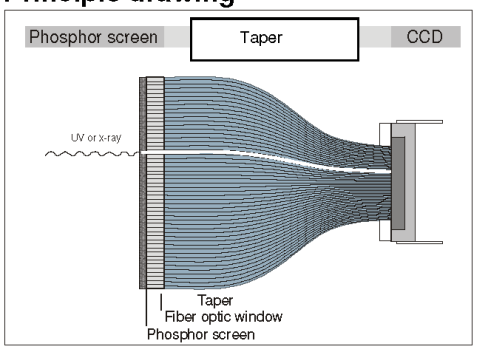
Customised versions are available on request (please specify the radiation to be detected):

- type and thickness of coating
- additional layers (e.g. Alu, ITO)
- type and thickness of input window or "open face"
- camera electronics
- cooling (for certain types)
- vacuum interface
- field of view

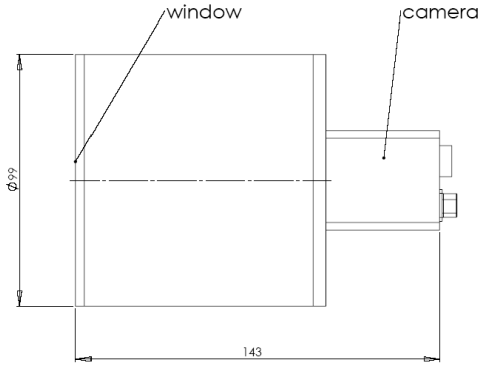
For customised versions please contact us. Based on our engineering and production facilities special versions can be realised.



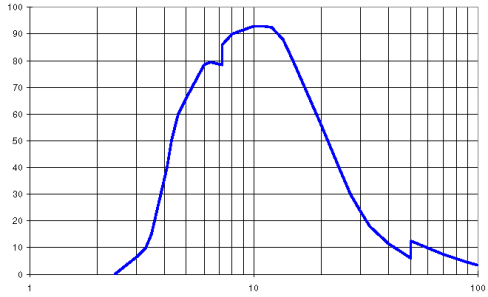
Principle drawing



Dimensions HR75 X-Ray



Quantum Efficiency (%)



Photon Energy (keV) [Quantum efficiency of P 43 (phosphor quantity: 25 mg/cm^2, layer thickness: ca. 55 µm)]
P43 is most recommended for a broad x-ray spectrum and UV radiation of a wavelength ≤ 250 nm.