Atomic and nuclear physics

X-ray physics

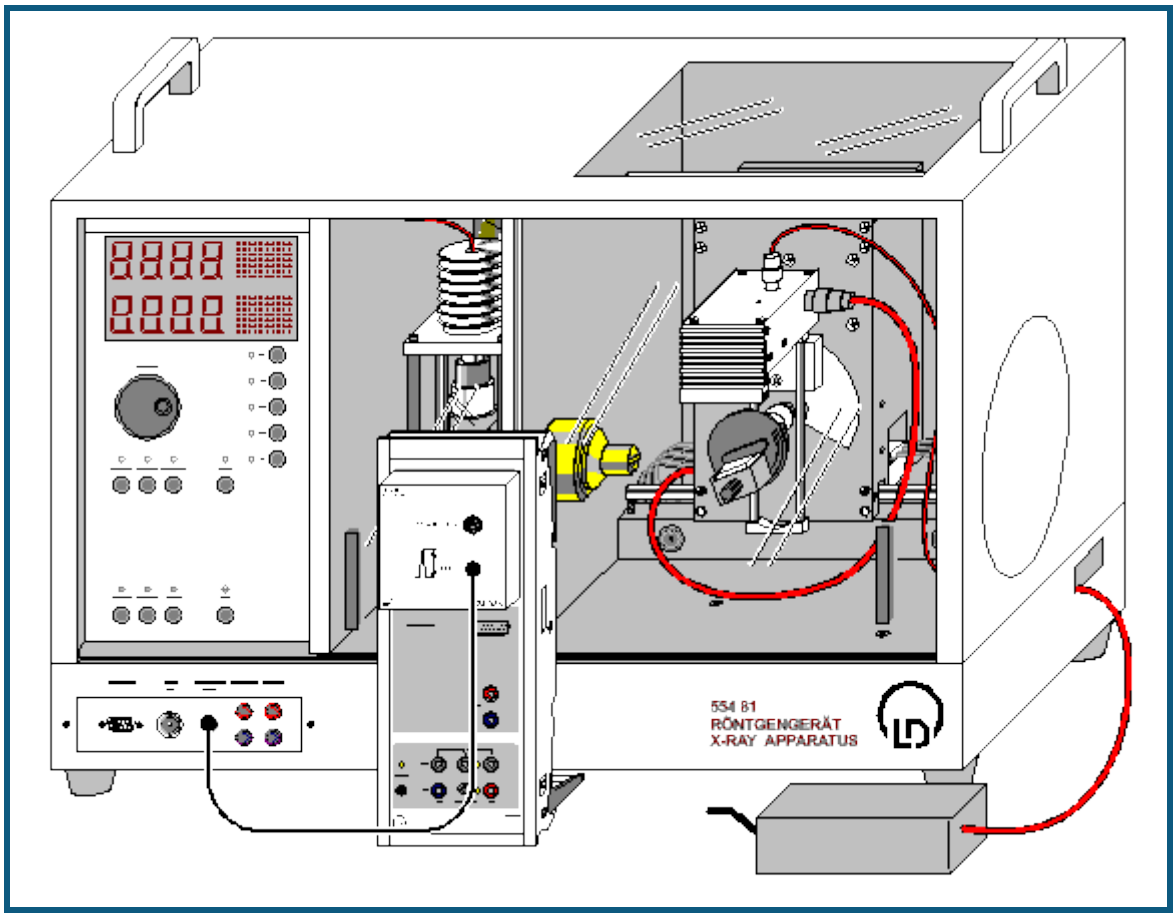
X-ray energy spectroscopy

Energy-resolved Bragg reflection in different orders of diffraction

Description from CASSY Lab 2

For loading examples and settings, please use the CASSY Lab 2 help.

Energy dispersive Bragg reflection into different orders of diffraction



can also be carried out with Pocket-CASSY

Safety notes

The X-ray apparatus fulfils all regulations on the design of an X-ray apparatus and fully protected device for instructional use and is type approved for school use in Germany (BfS 05/07 V/Sch RöV or NW 807 / 97 Rö).

The built-in protective and shielding fixtures reduce the dose rate outside the X-ray apparatus to less than 1 µSv/h, which is of the order of magnitude of the natural background radiation.

- Before putting the X-ray apparatus into operation, inspect it for damage and check whether the voltage is switched off when the sliding doors are opened (See instruction sheet of the X-ray apparatus).
- Protect the X-ray apparatus against access by unauthorized persons.

Avoid overheating of the anode in the X-ray tube.

- When switching the X-ray apparatus on, check whether the ventilator in the tube chamber starts rotating.

The goniometer is positioned solely by means of electric stepper motors.

- Do not block the target arm and the sensor arm of the goniometer and do not use force to move them.

Experiment description

According to a model first described by W.H. and W.L. Bragg in 1913, the regular arrangement of atoms in a crystal can be interpreted in such a way that they are arranged on parallel lattice planes.

If parallel x-rays hit the crystal these are elastically scattered by each of the atoms. If two conditions apply, the waves scattered at the individual atoms will interfere constructively.

These conditions are:

Incident angle α_1 = diffracted angle α_2 = α

and

$n \cdot \lambda = 2 \cdot d \cdot \sin \alpha$ (Bragg's condition)

with d the distance between the lattice planes and order of diffraction n being an integer number. The angle α applies relative to the lattice planes.

When recording the spectrum by means of an energy resolving detector, not the wavelengths, but the energies are recorded. For this case, Bragg's condition can be rewritten with $E = h\nu = hc/\lambda$ as follows:

$E_n = nhc / 2d\sin\alpha$

or

$E_n/n = hc / 2d\sin\alpha \approx 620\ pm \cdot keV / d\sin\alpha$

Here E_n is the energy of the x-ray radiation reflected in the order of diffraction n. It is apparent that with a fixed angle α the lowest energy E_1 occurs in the first order of diffraction. The ratio of the radiation energy in the order of diffraction n with the radiation energy in the first order E_n/E_1 corresponds to the order of diffraction. The ratio E_n/n is constant for every set of lattice planes.

Equipment list

1	Sensor-CASSY	524 010 or 524 013
1	CASSY Lab 2	524 220
1	MCA box	524 058
1	X-ray apparatus with goniometer	554 801 or 554 811
1	X-ray tube Cu	554 862 or 554 85
1	X-ray energy detector	559 938
1	HF cable, 1 m	501 02
1	PC with Windows XP/Vista/7/8	

Experiment setup (see drawing)

- If necessary exchange the Mo x-ray tube for the Cu x-ray tube (see operating instructions for the x-ray apparatus)
- Guide the connection cable for the table-top power supply through the empty channel of the x-ray apparatus and connect it to the mini-DIN socket of the x-ray energy detector.
- Secure the sensor holder with the mounted x-ray energy detector in the goniometer sensor arm
- Connect the signal output of the x-ray energy detector to the BNC socket SIGNAL IN of the x-ray apparatus by means of the BNC cable included
- Feed enough connection cable through to make complete movement of the sensor arm possible
- Connect Sensor-CASSY to the computer and connect the MCA box
- Connect the SIGNAL OUT output in the connection panel of the x-ray apparatus to the MCA box by means of the BNC cable.
- Set the distances between the slit aperture of the collimator and the axis of rotation as well as between the axis of rotation and the window of the x-ray energy detector both to 5 to 6 cm
- Lay the NaCl crystal from the scope of delivery of the X-ray apparatus on the target stage and clamp it gently.
- Press the COUPLED pushbutton, and, using the ADJUST knob, adjust a target angle of 4.5° manually.

Remark

For the experiment, the Cu x-ray tube is inserted, because, due to its thinner window, the radiation provided covers a wider energy range: approx. 5 to 35 keV at the high voltage 35 kV. The crystal angles are selected in such a way that the bremsstrahlung and not the characteristic lines are reflected by the crystal. Otherwise the much larger intensity in the characteristic line can considerably falsify the intensity ratio of various orders of diffraction.

Carrying out the experiment

☐ Load settings

- Connect the table-top power supply to the mains (after approx. 2 min the LED will glow green and the x-ray energy detector will be ready for use)
- Set the tube high voltage U = 35 kV, emission current I = 1.00 mA and switch the high voltage on
- Record the calibration spectrum (start the spectrum recording with ⏱)
- Set the emission current to I = 0.40 mA
- Record the spectra for the target angles 5°, 10°, 15° and 20°

Energy calibration

The X-rays to be measured produce additional fluorescence X-rays in the housing of the Si-PIN photodiode of the X-ray energy detector, which are also registered. In the primary spectrum therefore, in addition to the peaks of the reflected radiation, the gold (Au) and the silver (Ag) lines are also to be expected. With the help of these lines the energy calibration of the spectra can be carried out.

Through the scatter of the Cu K_α-line of the primary spectrum of the x-ray tube, on the left-hand side a peak occurs at E=8.1 keV. The subsequent smaller peaks at E=9.7 keV, 11.4 keV and 22.2 keV have been caused by the fluorescence of the housing (Au L_α, L_β and Ag K_α) and the large peak on the right-hand side is the reflected radiation (Bragg's condition).

- Select spectrum N_1 (4.5°)
- Open in the Settings EA (right mouse button) the Energy calibration, select **Global for all spectra of this input** and enter on the right-hand side the energies of the Au L_α-line (9.71 keV) and of the Ag K_α-line (22.16 keV).
- In the context menu of the diagram select Calculate peak center, mark the Au L_α-line and enter the result in the left-hand side of the Energy calibration (e.g. with drag & drop from the status line)
- Then determine the center for the Ag K_α-line and also enter it on the left-hand side.
- Switch the display to energy (e.g. with Drag & Drop of E_A into the diagram)

Remark

In order to excite the silver K-lines in the housing material, the energy of the reflected radiation must exceed the K-edge of silver (25.52 keV). For this reason the energy calibration is carried out on the spectrum for α=4.5°.

Evaluation

For confirmation that the remaining peaks associated with the various orders of diffraction belong to the same set of lattice planes, the ratios of the peak energies must be analyzed.

For the determination of the peak energies as a function of the scatter angle

- Select energy spectrum (5°, 10°, 15° and 20°).
- In the context menu of the diagram select Calculate peak center and mark the desired peak
- Read the determined peak positions from the status line and enter as E_n=1, E_n=2, E_n=3 or E_n=4 together with the angle α into the **Energy** diagram (click with mouse) (e.g. by drag & drop from the status line)

In the **Order** diagram a table is formed in which the ratios E_n/E_1 of the radiation energy in the order of diffraction n with the first order radiation energy are given. They have approximately integer values which confirms the assumption that they belong to different orders of diffraction from the same set of lattice planes.

In the **Lattice plane spacing** diagram the lattice plane spacing d was calculated for each crystal angle from the determined values E_n/n. It becomes apparent that the diffraction for all angles was observed from the same set of lattice planes with a spacing of d ≈ 280 pm. By comparing with the data given in the literature for NaCl (grid constant 564 pm) it becomes apparent that this is the diffraction from the (200) lattice planes (d = 282 pm).

Additional information

For a Bragg spectrum recorded using a Geiger-Müller counter as the detector, the photons which are reflected in the various orders of diffraction are counted together. A spectrum recorded in this way is therefore a combination of several components which cannot easily be separated. In the 20° spectrum it is particularly clear how incorrect the statement about the intensity of radiation would be if only the first order of diffraction were taken into account and the existence of higher orders of diffraction were ignored.