

## Energy-resolved Bragg reflection in different orders of diffraction

### Objects of the experiments

- Demonstration of the simultaneous first and higher order diffraction on a crystal.
- Investigating the ratio of the energies of the X-rays in different orders of diffraction.
- Determining the interplanar crystal spacing of the NaCl crystal.

### Principles

According to a consideration first carried out by W.H. and W.L. Bragg in 1913, the regular arrangement of atoms in a crystal can be interpreted such that they are arranged on parallel lattice planes (see also LD Physics Leaflet P7.1.2.1).

When parallel X-rays impinge on a crystal, they are scattered elastically on each atom. If two conditions are fulfilled, the waves scattered on the individual atoms interfere constructively. These conditions read:

angle of incidence  $\alpha_1 =$  angle of scattering  $\alpha_2 = \alpha$

and

$$n \cdot \lambda = 2 \cdot d \cdot \sin \alpha, \quad (I)$$

where  $d$  is the interplanar crystal spacing and the order of diffraction  $n$  is an integer. The angle  $\alpha$  is measured with respect to the lattice planes.

Eq. (I) is derived by considering the rays scattered on two lattice planes. In Fig. 1 (a), the incoming rays are denoted by 1 and 2 and the scattered rays by 1' and 2'. The rays 1' and 2' interfere constructively if the path difference  $\Delta L = AB + BC$  corresponds to an integer multiple of the wavelength. As the angle  $\angle ADB = \angle BDC = \alpha$ , the path difference is  $\Delta L = 2d \sin \alpha$ .

In Fig. 1, the situation is shown for three orders of diffraction: Picture (a) corresponds to the first order with  $\Delta L = 1 \cdot \lambda$ , picture (b) to the second order with  $\Delta L = 2 \cdot \lambda$  and picture (c) to the third order with  $\Delta L = 3 \cdot \lambda$ .

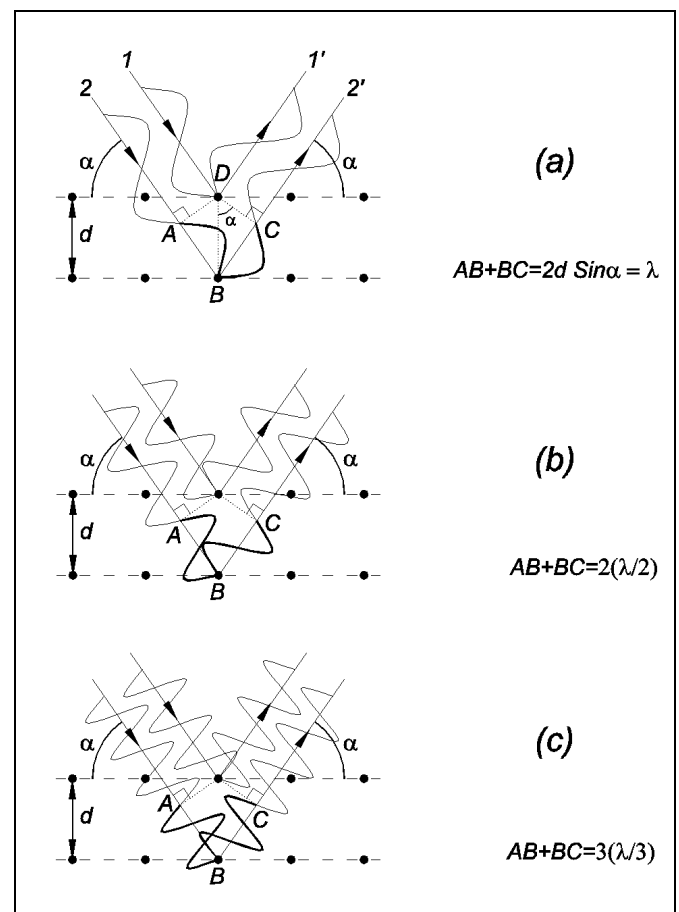


Fig. 1: Schematic illustration of Bragg reflection and the derivation of the Bragg condition. (a): first order of diffraction ( $n=1$ ); (b): second order ( $n=2$ ); (c): third order ( $n=3$ ).

If the spectrum is recorded with the aid of an energy resolving detector, the energies are measured rather than the wavelengths. For this case, the Bragg condition Eq. (I) can be cast into the following form, taking into account the relation  $E = h\nu = hc/\lambda$ :

$$E_n = n \frac{hc}{2d \sin \alpha} \quad (\text{II})$$

Here  $E_n$  is the energy of the X-rays reflected in the order of diffraction  $n$ . Obviously, the smallest energy  $E_1$  occurs in the first order at a fixed angle  $\alpha$ . The ratio of the energy of radiation diffracted in the order of diffraction  $n$  to the energy diffracted in the first order  $E_n/E_1 = n$  is equal to the order of diffraction.

It follows from Eq. (II) that the ratio  $E_n/n$  is constant for any set of lattice planes:

$$\frac{E_n}{n} = \frac{hc}{2d \sin \alpha} \approx \frac{620 \text{ pm} \cdot \text{keV}}{d \sin \alpha} \quad (\text{III})$$

With the aid of the equation, the interplanar spacing  $d$  of the lattice can be calculated from the energies.

### Apparatus

1 X-ray apparatus with goniometer	554 811
1 X-ray tube Cu	554 85
1 X-ray energy detector	559 938
1 Sensor-CASSY	524 010
1 MCA box	524 058
1 CASSY Lab	524 200
1 BNC cable, 1 m	501 02
1 PC with Windows 98/NT or higher version	

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 (NW 807 / 97 Rö).

The built-in protective and shielding fixtures reduce the dose rate outside the X-ray apparatus to less than  $1 \mu\text{Sv/h}$ , 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 high 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 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 sensor arm and do not use force to move them.

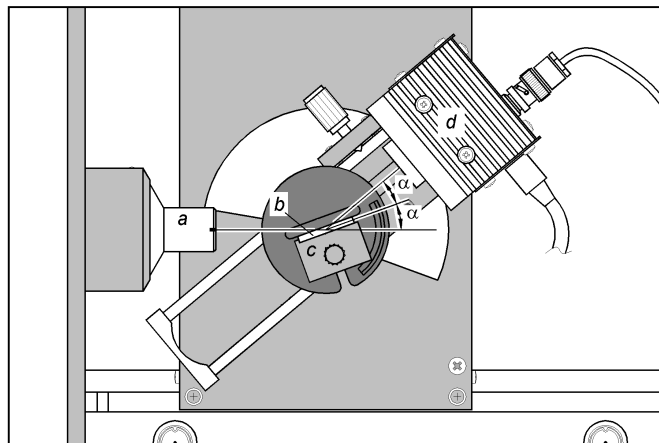


Fig. 2: Setup of the experiment: a – collimator, b – NaCl crystal, c – target stage, d – detector.

### Setup


The experimental setup is illustrated in Fig. 2.

- If necessary, replace the X-ray tube Mo with the X-ray tube Cu (see instruction sheet for the X-ray apparatus).
- Guide the connection cable of the table power supply through the empty duct of the X-ray apparatus, and connect it to the Mini-DIN socket of the X-ray energy detector.
- Fasten the assembly of the X-ray energy detector and the sensor holder in the sensor arm of the goniometer.
- Use the BNC cable supplied with the X-ray energy detector to connect the signal output of the detector to the BNC socket SIGNAL IN of the X-ray apparatus.
- Push a sufficient length of the connection cable through the duct so that the sensor arm can perform a complete movement.
- Connect the Sensor-CASSY to the computer, and plug in the MCA box.
- Use a BNC cable to connect the output SIGNAL OUT on the terminal panel of the X-ray apparatus to the MCA box.
- Adjust the distances between the slit of the collimator and the axis of rotation and between the axis of rotation and the entrance aperture of the X-ray energy detector each to 5-6 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  $4.5^\circ$  manually.

### Remark:

For this experiment the X-ray tube Cu is used because it provides the radiation in a wide energy range due to the thinner window: approx. 5 to 35 keV at a high voltage of 35 kV. The crystal angles are chosen such that only bremsstrahlung, but not the characteristic lines are reflected on the crystal. Otherwise the intensity of the characteristic line, which is much greater, could distort the intensity ratios of different orders of diffraction considerably.

## Carrying out the experiment

- Connect the table power supply to the mains (after approx. 2 minutes the LED of the X-ray energy detector shines green indicating that it is ready for operation).
- Call CASSY Lab, and select the measuring parameters "Multichannel Measurement, 256 Channels, Negative Pulses, Gain = -2.5, Measuring Time = 120 s".
- Select the tube high voltage  $U = 35$  kV and the emission current  $I = 1$  mA, and switch the high voltage on.
- Record the calibration spectrum (start recording the spectrum with  or with the F9 key).
- Adjust an emission current  $I = 0.4$  mA.
- Record spectra for the target angles  $5^\circ$ ,  $10^\circ$ ,  $15^\circ$  and  $20^\circ$ .
- Store the entire measurement using an appropriate name.

## Measuring example

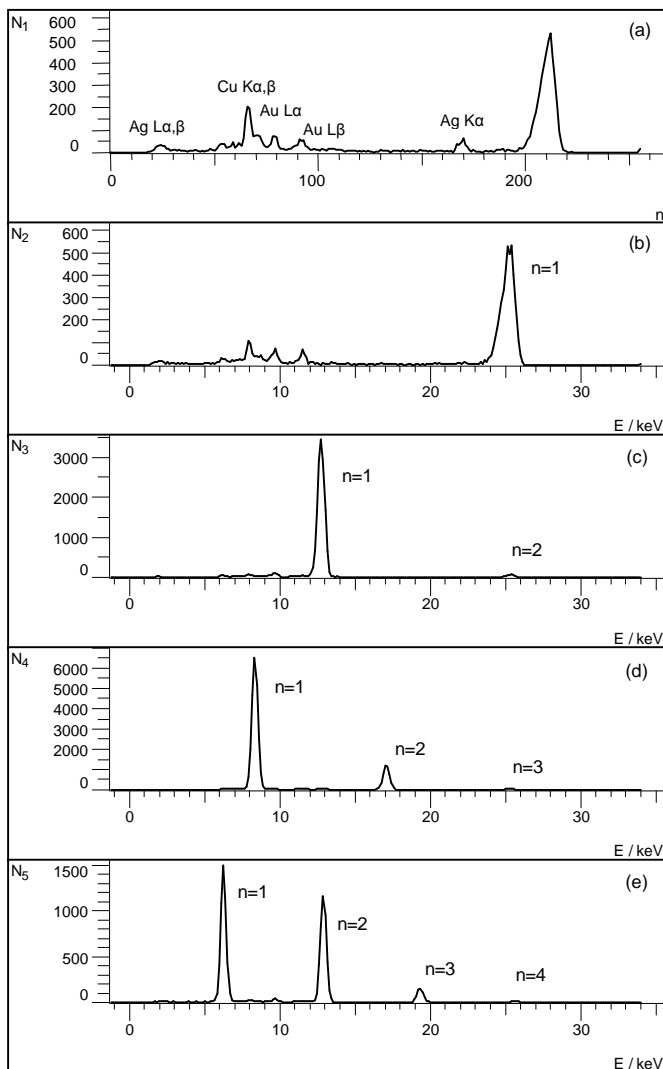


Fig. 3: Spectra of the X-rays reflected in the Bragg arrangement. Picture (a): crystal angle  $\alpha = 4.5^\circ$ ; (b):  $5^\circ$ ; (c):  $10^\circ$ ; (d):  $15^\circ$ ; (e):  $20^\circ$ . The orders of diffraction are indicated.

Fig. 3 shows the X-ray spectra recorded in the Bragg arrangement at the crystal angles  $\alpha = 4.5^\circ$ ,  $5^\circ$ ,  $10^\circ$ ,  $15^\circ$  and  $20^\circ$ . Obviously the higher orders are present in the spectrum apart from the 1st order (peak with the lowest energy). The small peaks at  $E = 9.7$  keV, 11.4 keV and 22.2 keV are caused by fluorescence of the housing ( $Au$   $L\alpha$ ,  $L\beta$  and  $Ag$   $K\alpha$ , respectively) and the peak at  $E = 8.1$  keV is due to scattering of the  $Cu$   $K\alpha$  line from the primary spectrum of the X-ray tube.

## Evaluation and results

### a) Energy calibration of the spectra

The X-rays to be measured produce additional fluorescence X-rays in the housing of the Si-PIN photodiode, which are also detected. Therefore the gold (Au) and silver (Ag) lines are to be expected in the primary spectrum apart from the peaks of the reflected radiation (see Fig. 3 a). With the aid of these lines, the energy calibration of the spectra can be carried out.

- Select the spectrum N1.
- Open the "Energy Calibration" dialog window with the shortcut Alt+E, select "Global Energy Calibration" and enter the energies of the Au  $L\alpha$  line (9.71 keV) and the Ag  $K\alpha$  line (22.16 keV).
- Select the menu item "Other Evaluations"  $\rightarrow$  "Calculate Peak Center" in the pop-up menu of the diagram window, mark the Au  $L\alpha$  line, and enter the result in the "Energy Calibration" dialog window.
- Then determine and enter the peak center of the Ag  $K\alpha$  line.

#### Remark:

In order to excite the K lines of silver in the housing material, the energy of the primary radiation has to exceed the K absorption edge of silver (25.52 keV). For this reason, the energy calibration is carried out on the spectrum with  $\alpha = 4.5^\circ$ .

### b) Confirming the peak origin

In order to confirm that the peaks in Fig. 3 are associated with the different orders of diffraction on the same set of lattice planes, the ratios of the peak energies have to be analysed.

*Determining the energy as a function of the scattering angle:*

- Select the energy spectrum.
- Select the menu item "Other Evaluations"  $\rightarrow$  "Calculate Peak Center" in the pop-up menu of the diagram window, and mark the region of the desired peak.
- Enter the peak center determined and the corresponding order of diffraction in a table (see Table 1).

Table 1: Energies  $E_n$  for different crystal angles  $\alpha$  and orders of diffraction  $n$ 

$\alpha$	$E_n$			
	$n = 1$	$n = 2$	$n = 3$	$n = 4$
5°	25.13 keV $E_n / E_1 = 1$			
10°	12.72 keV $E_n / E_1 = 1$	25.35 keV $E_n / E_1 = 1.99$		
15°	8.33 keV $E_n / E_1 = 1$	17.07 keV $E_n / E_1 = 2.05$	25.34 keV $E_n / E_1 = 3.04$	
20°	6.25 keV $E_n / E_1 = 1$	12.87 keV $E_n / E_1 = 2.06$	19.30 keV $E_n / E_1 = 3.09$	25.63 keV $E_n / E_1 = 4.10$

In Table 1 the ratios  $E_n / E_1$  of the radiation energy in the order  $n$  to the radiation energy in the first order are also listed. They have approximately integer values which, taking into account Eq. (II), confirms that they are associated with different orders of diffraction on the same set of lattice planes.

### c) Determining the interplanar spacing of crystal

According to Eq. (III), the interplanar spacing  $d$  of the  $\text{NaCl}$  crystal used can be obtained from the ratio  $E_n / n$ .

Table 2: Numerical example of the determination of the interplanar spacing  $d$  according to Eq. (III)

$\alpha$	$E_n / n, \text{ keV}$				$\langle E_n / n \rangle,$ keV	$d,$ pm
	$n = 1$	$n = 2$	$n = 3$	$n = 4$		
5°	25.13				25.13	283
10°	12.72	12.67			12.69	281
15°	8.33	8.53	8.45		8.44	284
20°	6.25	6.43	6.43	6.41	6.38	284

The values of  $d$  given in Table 2 have been calculated for each crystal angle from the mean values  $\langle E_n / n \rangle$  given in Table 1. Obviously the diffraction was observed at all angles on a set of lattice planes with the spacing  $d \approx 280$  pm. Comparison with data quoted in the literature for  $\text{NaCl}$  (lattice constant  $a_{\text{NaCl}} = 564.0$  pm) shows that the observed diffraction occurred on the (200) lattice planes ( $d_{200} = a_{\text{NaCl}} / 2 = 282$  pm).

### Supplementary information

If the Bragg reflection is recorded with a Geiger-Müller counter tube as a detector, the photons reflected in different orders of diffraction are counted *together*. A spectrum recorded this way thus contains several components, which cannot be simply separated. From the picture (e) in Fig. 3 it is particularly obvious how flawed a statement on the intensity of the radiation is if only the first order is taken into account and if the presence of higher orders is neglected.