Atomic and Nuclear Physics

X-ray physics
Physics of the atomic shell

LD
Physics
*Leaflets*

P6.3.3.1

*Bragg reflectio*n: diffraction of x-rays at a monocrystal

<div style="background:gray">

Objects of the experiment

■ To investigate Bragg reflection at an NaCl monocrystal using the characteristic x-ray radiation of molybdenum.

■ To determine the wavelength for the characteristic K_α and K_β x-ray radiation of molybdenum.

■ To confirm Bragg′s law of reflection.

■ To verify the wave nature of x-rays.

</div>
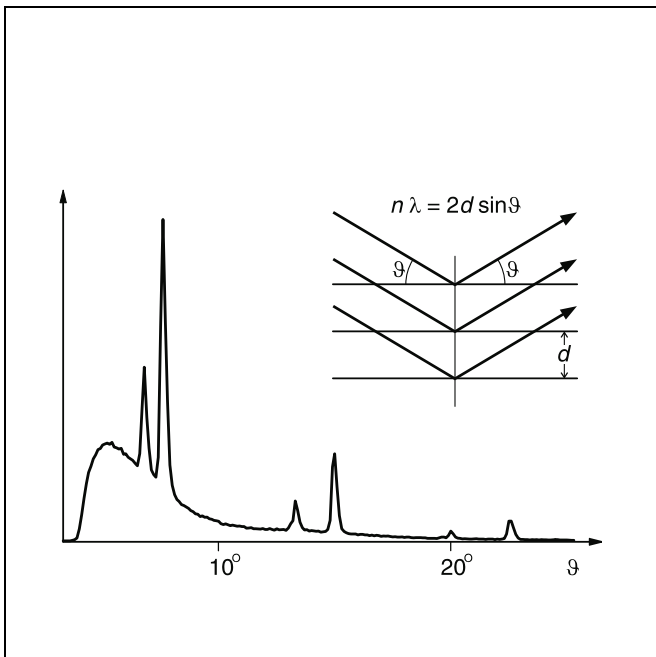
Principles

In 1913, *H. W.* and *W. L. Bragg* realized that the regular arrangement of atoms and/or ions in a crystal can be understood as an array of lattice elements on parallel lattice planes. When we expose such a crystal to parallel x-rays, additionally assuming that these have a wave nature, then each element in a lattice plane acts as a "scattering point", at which a spherical wavelet forms. According to *Huygens,* these spherical wavelets are superposed to create a "reflected" wavefront. In this model, the wavelength λ remains unchanged with respect to the "incident" wave front, and the radiation directions which are perpendicular to the two wave fronts fulfill the condition "angle of incidence = angle of reflection".

Constructive interference arises in the rays reflected at the individual lattice planes when their path differences Δ are integral multiples of the wavelength λ.

$$\Delta = n \cdot \lambda \text{ with } n = 1, 2, 3, \dots \tag{I}$$

As Fig. 1 shows for two adjacent lattice planes with the spacing *d,* we can say for the path differences Δ_1 and Δ_2 of the incident and reflected rays with the angle ϑ:

$$\Delta_1 = \Delta_2 = d \cdot \sin \vartheta$$

so that the total path difference is

$$\Delta = 2 \cdot d \cdot \sin \vartheta. \tag{II}$$

(I) and (II) give us Bragg′s law of reflection:

$$n \cdot \lambda = 2 \cdot d \cdot \sin \vartheta \tag{III}$$

The angle ϑ is known as the glancing angle.

In this experiment, we verify Bragg′s law of reflection by investigating the diffraction of x-rays at an NaCl monocrystal in which the lattice planes are parallel to the cubic surfaces of the unit cells of the crystal. The lattice spacing *d* of the cubic

Fig. 1   Diagram of the reflection of x-rays at the lattice planes of a
        monocrystal.
        Δ_1, Δ_2: path differences,
        ϑ: glancing angle,
        d: spacing of lattice planes

face-centered NaCl crystal is half the lattice constant a_0. We
can thus say [1]

$2 \cdot d = a_0 = 564.02$ pm

The measurements are conducted using the built-in goniome-
ter of the x-ray apparatus (554 811). The x-rays are detected
using a GM counter tube (end-window counter) which is
swiveled in tandem with the NaCl crystal in a 2_ coupling with
respect to the incident light; this means that the counter tube
always advances by an angle which is twice that of the crystal
(cf. Fig. 2).

The x-ray radiation consists of the bremsstrahlung continuum
and several sharply defined lines which correspond to the
characteristic x-ray radiation of the Mo anode and which
originate in the K_α and K_β transitions of the molybdenum
atoms. This characteristic radiation is particularly suitable for
investigating Bragg's law. Its properties are known from the
literature [2] and summarized in table 1. Table 2 shows the
corresponding glancing angles at which the diffraction maxima
of the characteristic radiation are to be expected for scattering
at an NaCl monocrystal (d = 282.01 pm) up to the third
diffraction order.

Table 1: Energy E, frequency ν and wavelength λ of the char-
acteristic x-ray radiation of molybdenum (weighted mean
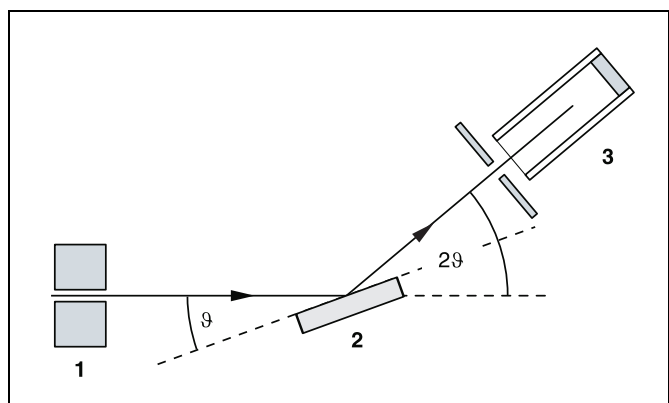values [1])

	$\dfrac{E}{\text{keV}}$	$\dfrac{\nu}{\text{EHz}}$	$\dfrac{\lambda}{\text{pm}}$
K_α	17.443	4.2264	71.080
K_β	19.651	4.8287	63.095

keV = 10^3 eV, EHz = 10^{18} Hz, pm = 10^{-12} m

Table 2: Glancing angle ϑ of the characteristic x-ray radiation
of molybdenum for diffraction at an NaCl monocrystal up to
the third order

n	$\vartheta(K_\alpha)$	$\vartheta(K_\beta)$
1	7.24°	6.42°
2	14.60°	12.93°
3	22.21°	19.61°

Fig. 2   Diagram showing the principle of diffraction of x-rays at a
        monocrystal and 2ϑ coupling between counter-tube angle
        and scattering angle (glancing angle)
        1 collimator, **2** monocrystal, **3** counter tube

General remarks

In principle, you can conduct measurements in both manual scan and autoscan modes of the x-ray apparatus (see the Instruction Sheet of the x-ray apparatus). You can record the measured values manually by reading the values from the display field and writing them in a table, using a chart recorder or via a PC.

The fastest and most convenient measurement is in autoscan mode with simultaneous registration of measured values and subsequent evaluation on a Windows 9x/NT PC. This type of measurement is described in your Instruction Sheet.

The data is transmitted to the PC via the RS–232 serial interface on the x-ray apparatus. The software "X-ray Apparatus", supplied with the device, enables you to record, display and evaluate the data stream supplied by the x-ray apparatus. The program contains detailed online help which you can access by pressing F1. Please refer to the Instruction Sheet of the x-ray apparatus for details on installing the software.

The Instruction Sheet also describes recording data under Windows 3.1.

Setup

Setting up the Bragg configuration:

Fig. 3 shows some important details of the experiment setup. Specifically, you need to carry out the following steps (see also Instruction Sheet of x-ray apparatus):

– Mount the collimator in the collimator mount **(a)** (note the guide groove).
– Attach the goniometer to the guide rods **(d)** in such a way that the distance s_1 between the slit diaphragm of the collimator and the target arm is approx. 5 cm. Connect the ribbon cable **(c)** for controlling the goniometer.
– Remove the cap of the end-window counter, insert the end-window counter in the sensor seat **(e)** and connect the counter tube lead to the socket marked GM-Tube.

Fig. 3   Experiment setup in Bragg configuration

– Adjust the sensor seat **(b)** until the distance s_2 between the target arm and the slit diaphragm of the sensor seat is approx. 6 cm.
– Attach the target holder with target stage **(f)**.
– Loosen knurled screw **(g)**, lay the NaCl crystal flat on the target stage, carefully raise the stage as far at it will go and then tighten the knurled screw with care (press against the screw lightly to prevent it from stripping).
– Adjust the zero position of the goniometer measuring system as necessary (see Instruction Sheet of x-ray apparatus).

Notes:

NaCl crystals are hygroscopic and fragile. Store the crystals in a dry place. Avoid mechanical stresses on the crystal; handle the crystal by the short faces only.

If the counting rate is too low, you can reduce the distance s_2 between the target and the sensor somewhat. However, this distance must not be too small, as otherwise the angular resolution of the goniometer is no longer great enough to separate the characteristic K_α and K_β lines.

Preparing a PC-based measurement:

– Connect the RS–232 output to the serial interface on the PC (usually COM1 or COM2) using the 9-pin V.24 cable (included with the x-ray apparatus).
– If you have not already done so, install the software "X-ray Apparatus" under Windows 9x/NT (see Instruction Sheet of x-ray apparatus) and select the desired language.

Carrying out the experiment

– Start the program "X-ray Apparatus", check to make sure that the x-ray apparatus is properly connected and delete any existing measurement data by clicking the button [ ] or pressing F4.
– Set the x-ray high voltage U = 35.0 kV, emission current I = 1.00 mA, measuring time per angular step Δt = 10 s and angular step width $\Delta\beta$ = 0.1°.
– Press the COUPLED key on the device to enable 2ϑ coupling of the target and sensor; set the lower limit value of the target angle to 2° and the upper limit to 25°.
– Press the SCAN key to start the measurement and data transmission to the PC.
– When the measurement is finished, save the measurement series to a file under a suitable name using the button [ ] or F2.
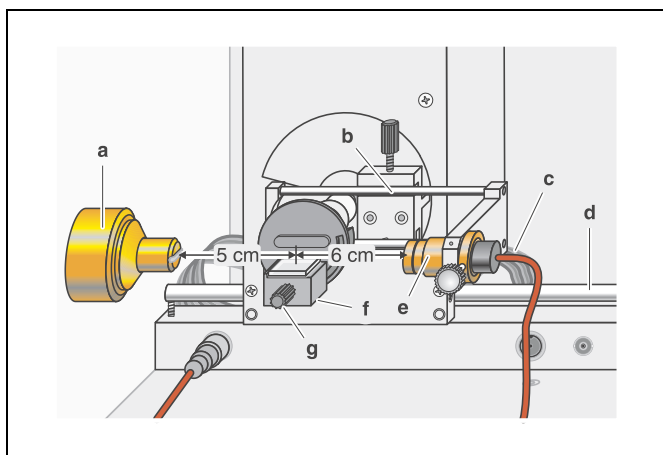
Measuring example

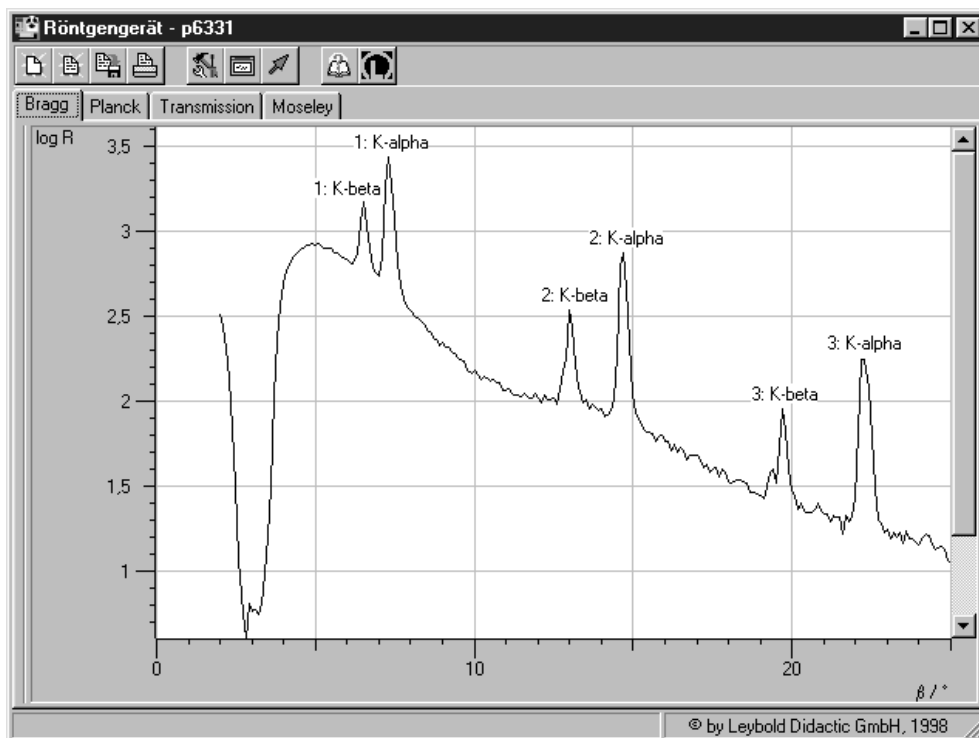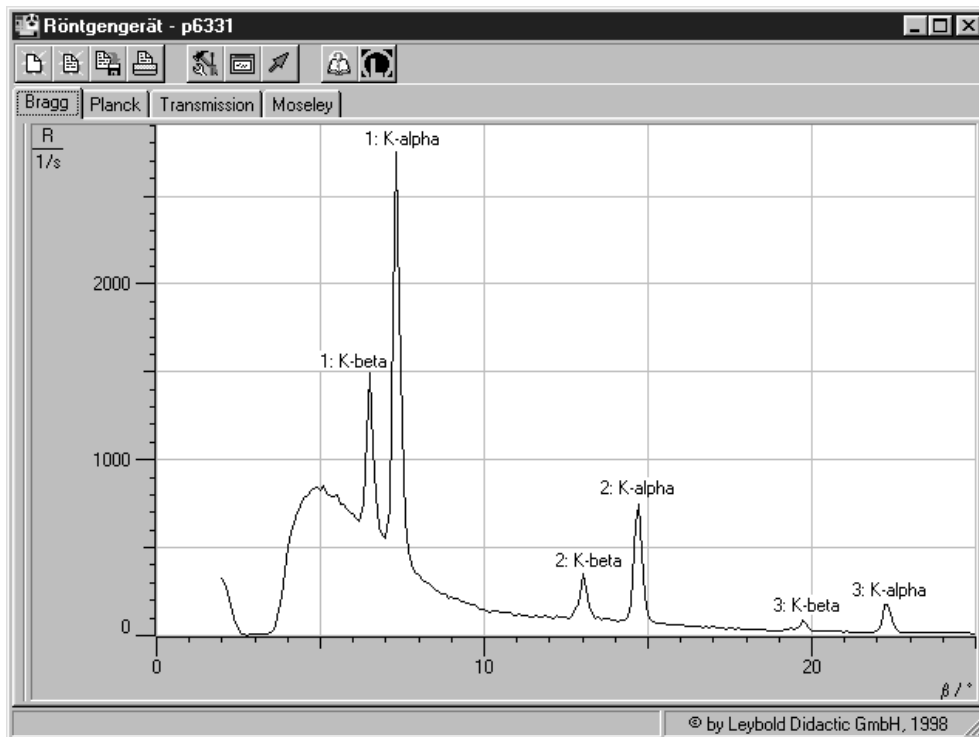Fig. 4 shows the measured diffraction spectrum.

Fig. 4 Diffraction spectrum of x-ray radiation for *Bragg* reflection to the third order
at an NaCl monocrystal
Top: linear representation of counting rate *R*
Bottom: logarithmic representation of counting rate *R*
Parameters of x-ray tube: *U* = 35 kV and *I* = 1 mA

Evaluation

– Access the evaluation functions of the software "X-ray Apparatus" by clicking the right-hand mouse button and select the command "Calculate Peak Center".

– Using the left mouse button, mark the "entire width" of the peaks; if desired, insert the calculated peak center β and the peak width σ in the diagram with Alt+T and note the center as the glancing angle in the measurement table (see tables 3 and 4).

– Save your measurements and evaluations to a suitably named file with the button ⌷ or by pressing F2.

– Using the glancing angle ϑ and the lattice plane spacing d = 282.01 pm, calculate the wavelength λ using Bragg's law of reflection (IV) (see tables 3 and 4).

– Find the mean values for the individual diffraction orders of the measured wavelengths (see table 5).

Table 3: Measured glancing angles of the Mo K_α line and the calculated wavelengths λ for the first through third diffraction orders

n	$\vartheta(K_\alpha)$	$\dfrac{\lambda(K_\alpha)}{pm}$
1	7.24°	71.08
2	14.60°	71.09
3	22.20°	71.04

Table 4: Measured glancing angles of the Mo K_β line and the calculated wavelengths λ for the first through third diffraction orders

n	$\vartheta(K_\beta)$	$\dfrac{\lambda(K_\beta)}{pm}$
1	6.42°	63.07
2	12.94°	63.15
3	19.58°	63.01

Table 5: Mean value and literature value [2] for the characteristic wavelength λ

	$\dfrac{\lambda(K_\alpha)}{pm}$	$\dfrac{\lambda(K_\beta)}{pm}$
Mean value	71.07	63.08
Literature value	71.08	63.09

Results

The close agreement of the experimentally determined wavelengths for the characteristic lines with the literature values in table 5 verify the validity of Bragg's law. This simultaneously confirms the wave nature of x-rays, as this property was assumed in the process of deducing this law.

Additional information

The characteristic K_α and K_β lines actually consist of multiple, adjacent discrete lines, which can be observed separately at higher diffraction orders (see Physics Leaflet P 6.3.3.4). Table 1 shows the weighted mean values of the respective individual lines from this substructure.

Literature

[1] Handbook of Chemistry and Physics, 52nd Edition (1971–72), The Chemical Rubber Company, Cleveland, Ohio, USA.

[2] C. M. Lederer and V. S. Shirley, Table of Isotopes, 7th Edition, 1978, John Wiley & Sons, Inc., New York, USA.