5.4.02–00 Characteristic X-rays of molybdenum

**What you need:**

- X-ray basic unit, 35 kV: 09058.99
- Goniometer for X-ray unit, 35 kV: 09058.10
- Plug-in module with Mo x-ray tube: 09058.60
- Counter tube, type B: 09005.00
- Lithium fluoride crystal, mounted: 09056.05
- Potassium bromide crystal, mounted: 09056.01

**Recording equipment:**

- XYt recorder: 11416.97
- Connecting cable, $l = 100$ cm, red: 07363.01
- Connecting cable, $l = 100$ cm, blue: 07363.04
- Software x-ray unit, 35 kV: 14407.61
- RS232 data cable: 14602.00
- PC, Windows® 95 or higher

**What you can learn about ...**

- X-ray tube
- Bremsstrahlung
- Characteristic radiation
- Energy levels
- Crystal structures
- Lattice constant
- Absorption
- Absorption edges
- Interference
- The Bragg equation
- Order of diffraction

**Tasks:**

1. The intensity of the X-rays emitted by the molybdenum anode at maximum anode voltage and anode current is to be recorded as a function of the Bragg angle, using an LiF monocrystal as analyzer.
2. Step 1 is to be repeated using the KBr monocrystal as analyzer.
3. The energy values of the characteristic molybdenum lines are to be calculated and compared with the energy differences of the molybdenum energy terms.
Related topics
X-ray tube, bremsstrahlung, characteristic radiation, energy levels, crystal structures, lattice constant, absorption, absorption edges, interference, the Bragg equation, order of diffraction.

Principle
Spectra of X-rays from a molybdenum anode are to be analyzed by means of different monocrystals and the results plotted graphically. The energies of the characteristic lines are then to be determined from the positions of the glancing angles for the various orders of diffraction.

Equipment
- X-ray basic unit, 35 kV 09058.99 1
- Goniometer for X-ray unit, 35 kV 09058.10 1
- Plug-in module with Mo x-ray tube 09058.60 1
- Counter tube, type B 09005.00 1
- Lithium fluoride crystal, mounted 09056.05 1
- Potassium bromide crystal, mounted 09056.01 1

Recording equipment:
- XYt recorder 11416.97 1
- Connecting cable, l = 100 cm, red 07363.01 2
- Connecting cable, l = 100 cm, blue 07363.04 2
- Software x-ray unit, 35 kV 14407.61 1
- RS232 data cable 14602.00 1
- PC, Windows® 95 or higher

Tasks
1. The intensity of the X-rays emitted by the molybdenum anode at maximum anode voltage and anode current is to be recorded as a function of the Bragg angle, using an LiF monocrystal as analyzer.
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Set-up and procedure
Set up the experiment as shown in Fig. 1. Fix the diaphragm tube with 1 mm diameter aperture in the X-ray outlet tube. With the X-ray basic unit switched off, connect the goniometer and the counter tube to the appropriate sockets in the base plate of the experimenting area. Set the goniometer block with mounted analyzing crystal to the middle position and the counter tube to the right stop.

The following settings are recommended for the recording of the spectra:
- Auto and Coupling mode
- Gate time 2 s; Angle step width 0.1°
- Scanning range 4°-65° using the LiF monocrystal, and 3°-30° using the KBr monocrystal
- Anode voltage $U_A = 35$ kV; Anode current $I_A = 1$ mA

When the spectra are to be recorded with an XY recorder, connect the Y axis to the analog output (Imp/s) of the X-ray unit and, correspondingly, the X input to the analog output for the angular position of the crystal (select the analog signal for the crystal angle with the selection button for this output). When a PC is to be used for recording purposes, connect it via the SUB-D socket of the X-ray basic unit.

Note
Never expose the counter tube to primary radiation for a longer length of time.
Theory and evaluation

When electrons of high energy impinge on the metallic anode of an X-ray tube, X-rays with a continuous energy distribution (the so-called bremsstrahlung) are produced. X-ray lines whose energies are not dependent on the anode voltage and which are specific to the anode materials, the so-called characteristic X-ray lines, are superimposed on the continuum. They are produced as follows: An impact of an electron on an anode atom in the K shell, for example, can ionize that atom. The resulting vacancy in the shell is then filled by an electron from a higher energy level. The energy released in this de-excitation process can then be transformed into an X-ray which is specific for the anode atom.

Fig. 2 shows the energy level scheme of a molybdenum atom. Characteristic X-rays produced from either the L —> K or the M —> K transitions are called K<sub>a</sub>- and K<sub>b</sub>- lines respectively. M<sub>1</sub> —> K and L<sub>1</sub> —> K transitions do not take place due to quantum mechanical selection rules. Accordingly, characteristic lines for Mo with the following energies are to be expected (Fig. 2):

\[ E_{K_a} = E_K - \frac{1}{2}(E_{L_2} + E_{L_3}) = 17426.8 \text{ eV} \]  
\[ E_{K_b} = E_K - E_{M_{2,3}} = 19589.8 \text{ eV} \]

K<sub>a</sub>* is used as the mean value of the lines K<sub>a1</sub> and K<sub>a2</sub>.

The analysis of polychromatic X-rays is made possible through the use of a monocrystal. When X-rays of wavelength \( \lambda \) impinge on a monocrystal under glancing angle \( \theta \), constructive interference after scattering only occurs when the path difference \( \Delta \) of the partial waves reflected from the lattice planes is one or more wavelengths (Fig. 3).

This situation is explained by the Bragg equation:

\[ 2d \sin \theta = n\lambda \]  
\((d = \text{the interplanar spacing}; \ n = \text{the order of diffraction})\)

If \( d \) is assumed to be known, then the energy of the X-rays can be calculated from the glancing angle \( \theta \), which is obtainable from the spectrum, and by using the following relationship:

\[ E = h \cdot f = hc/\lambda \]

On combining (3) and (2), we obtain:

\[ E = (n \cdot h \cdot c) / (2 \cdot d \cdot \sin \theta) \]

Planck’s constant \( h = 6.6256 \cdot 10^{-34} \text{Js} \)
Velocity of light \( c = 2.9979 \cdot 10^8 \text{m/s} \)
Lattice constant LiF (100) \( d = 2.014 \cdot 10^{-10} \text{m} \)
Lattice constant KBr (100) \( d = 3.290 \cdot 10^{-10} \text{m} \)
and the equivalent 1 eV = 1.6021 \cdot 10^{-19} \text{J}
Fig. 4 shows that well-defined lines are superimposed on the bremsstrahlung spectrum continuum. The angles at which these lines are positioned remain unaltered on varying the anode voltage. This indicates that these lines are characteristic molybdenum lines. The first pair of lines belongs to the first order of diffraction ($n = 1$). As the intensity decreases at higher order diffraction, only the $K_a$ line is visible for $n = 4$ and $n = 5$. A weak indication of the separation of the $K_a$-doublet can be observed for $n = 4$ and $n = 5$ (see experiment 5.4.07-00).

When the KBr monocrystal is used instead of the LiF monocrystal to analyze the molybdenum X-ray spectrum, Bragg scatterings are allowed up to an order of diffraction of 4 ($n = 4$) (Fig. 5). The structures which are additional to those in Fig. 4 result from the higher lattice constant of the KBr monocrystal. The energy values of the characteristic molybdenum X-ray lines are listed in the Table, as calculated using (4).

A variation of the evaluation is possible by using the calculated characteristic molybdenum X-ray lines from one spectrum in order to derive the corresponding lattice constant from the other spectrum.

The bremsstrahlung in Fig. 6 is subject to a noticeable drop in intensity in the direction of smaller angles at 8.2° and 16.5°. This drop coincides with the theoretically expected Brønsted-K absorption edge ($E_K = 13.474$ keV) in the 1st and 2nd order of diffraction. The K absorption edges of potassium, lithium and fluorine cannot be observed, since the intensity of the bremsstrahlung spectrum is too low in these energy regions. (For K and L absorption edges, refer to experiment 5.4.12-00).

Note

The atomic energy values were taken from the "Handbook of Chemistry and Physics", CRC Press Inc., Florida.

Table of results

<table>
<thead>
<tr>
<th>$\theta/°$</th>
<th>Line</th>
<th>$E_{exp}$/keV</th>
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<tr>
<td>LiF analyzer (Fig. 4)</td>
<td></td>
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<tr>
<td>$n = 1$</td>
<td>10.2</td>
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<tr>
<td>KBr analyzer (Fig. 5)</td>
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</table>

Taking the energy values from the Table, the mean values of the energies of the characteristic lines are: $E_{K_a} = 17.248$ keV and $E_{K_b} = 19.268$ keV. Both of these experimental values correspond with literature values (see (1) and Fig. 2).
Characteristic X-rays of molybdenum