EXPERIMENT 1: CHARACTERISTIC X-RAYS OF COPPER

Related Topics
X-ray tubes, bremsstrahlung, characteristic X-radiation, energy levels, crystal structures, lattice constant, absorption of X-rays, absorption edges, interference, and Bragg's law.

Principle
An X-ray tube with a copper anode generates X-radiation that is selected with the aid of a monocrystal as a function of the Bragg angle. A Geiger-Müller counter tube measures the intensity of the radiation. The glancing angles of the characteristic X-ray lines are then used to determine the energy.

Tasks
- Analyse the intensity of the copper X-radiation as a function of the Bragg angle and with the aid of a LiF monocrystal.
- Analyse the intensity of the copper X-radiation as a function of the Bragg angle and with the aid of a KBr monocrystal.
- Determine the energy values of the characteristic X-rays of copper and compare them with the values that were determined based on the corresponding energy-level diagram.

Theory and Evaluation
When electrons impinge on the metallic anode of the X-ray tube with a high level of kinetic energy, X-rays with a continuous energy distribution (the so-called bremsstrahlung) are produced. The spectrum of the bremsstrahlung is superimposed by additional discrete lines. If an atom of the anode material is ionised on the K shell following an electron impact, an electron from a higher shell can take up the free place while emitting an X-ray quantum. The energy of this X-ray quantum corresponds to the energy difference of the two shells that are involved in this process. Since this energy difference is atom-specific, the resulting radiation is also called characteristic X-radiation.

Figure 8 shows the energy-level diagram of a copper atom. Characteristic X-radiation that is produced following a transition from the L shell to the K shell is called $K_{\alpha}$ radiation, while the radiation that is produced following a transition from the M shell to the K shell is called $K_{\beta}$ radiation ($M_1 \rightarrow K$ and $L_1 \rightarrow K$ transitions are not allowed due to quantum-mechanical selection rules). $\Delta l = \pm 1$ and $\Delta l = 0, \pm 1$ selection rules for the dipole radiation ($l = $ orbital angular momentum, $j = $ total angular momentum)
The characteristic X-ray lines of copper have the following energy levels (Fig. 8):

\[ E_{K\alpha}^* = E_K - \frac{1}{2} (E_{L2} + E_{L3}) = 8.038 \text{ keV} \]
\[ E_{K\beta} = E_K - E_{M2.3} = 8.905 \text{ keV} \]

\( E_{K\alpha}^* \) is the energetic mean value of the \( K\alpha_1 \) and \( K\alpha_2 \) lines.

The analysis of polychromatic X-rays is made possible through the use of a monocrystal. When X-rays of the wavelength \( \lambda \) impinge on the lattice planes of a monocrystal under the glancing angle \( \theta \), the rays that are reflected on the lattice planes interfere with each other in a constructive manner provided that their path difference \( \Delta \) corresponds to an integral multiple of the wavelength. In accordance with Figure 9, Bragg's law applies to constructive interference:

\[ 2d \sin \theta = n \lambda \]

\((d=\text{interplanar spacing}; \ n=1,2,3...\))

If the interplanar spacing \( d \) is known, the wavelength \( \lambda \) can be determined with the aid of the glancing angle \( \theta \). The energy of the radiation then results from:

\[ E = hf = \frac{hc}{\lambda} \]
When combining last two equation, we obtain:

\[ E = \frac{n \ h \ c}{2 \ d \ \sin\theta} \]

Planck's constant \( h = 6.6256 \times 10^{-34} \text{ Js} \)

Velocity of light \( c = 2.9979 \times 10^8 \text{ m/s} \)

Interplanar spacing LiF (200) \( d = 2.014 \times 10^{-10} \text{ m} \)

Interplanar spacing KBr (200) \( d = 3.290 \times 10^{-10} \text{ m} \)

Equivalent \( 1 \text{ eV} = 1.6021 \times 10^{-19} \text{ J} \)

**Equipment**

X-ray experiment unit, X-ray goniometer, X-ray plug-in unit with a copper X-ray tube, counter tube, Lithium Floride crystal, Potassium Bromide crystal.

![Fig.1: Experimental set-up for X-ray diffraction.](image)

**Set-up and Procedure**

Connect the goniometer and the Geiger-Müller counter tube to their respective sockets in the experiment chamber (see the red markings in Fig. 2). The goniometer block with the analyser crystal should be located at the end position on the right-hand side. Fasten the Geiger-Müller counter tube with its holder to the back stop of the guide rails. Do not forget to install the
Insert a diaphragm tube with a diameter of 2 mm into the beam outlet of the tube plug-in unit.

For calibration: Make sure, that the correct crystal is entered in the goniometer parameters. Then, select “Menu”, “Goniometer”, “Autocalibration”. The device now determines the optimal positions of the crystal and the goniometer to each other and then the positions of the peaks.
**Procedure**

- Connect the X-ray unit via the USB cable to the USB port of your computer (the correct port of the X-ray unit is marked in Figure 4).
- Start the "measure" program. A virtual X-ray unit will be displayed on the screen.
- You can control the X-ray unit by clicking the various features on and under the virtual X-ray unit. Alternatively, you can also change the parameters at the real X-ray unit. The program will automatically adopt the settings.
- Click the experiment chamber (see the red marking in Figure 5) to change the parameters for the experiment. Select the parameters as shown in Figure 6 for the LiF crystal. If you use the KBr crystal, select a start angle of $3^\circ$ and a stop angle of $75^\circ$.
- If you click the X-ray tube (see the red marking in Figure 5), you can change the voltage and current of the X-ray tube. Select the parameters as shown in Fig. 7.
- Start the measurement by clicking the red circle:

- After the measurement, the following window appears:

**Overview of the settings of the goniometer and X-ray unit:**
- 1:2 coupling mode
- Gate time 2 s; angle step width 0.1°
- Scanning range $4^\circ$-$55^\circ$ (LiF monocrystal) and $3^\circ$-$75^\circ$ (KBr monocrystal)
- Anode voltage $U_A = 35$ kV; anode current $I_A = 1$ mA
- Select the first item and confirm by clicking OK. The measured values will now be transferred directly to the “measure” software.
- At the end of this manual, you will find a brief introduction to the evaluation of the resulting spectra.

**Evaluation and results**

In the following section, the evaluation of the data is described based on example results. Your results may differ from the results given below.

*Task 1: Analyse the intensity of the copper X-radiation as a function of the Bragg angle and with the aid of a LiF monocrystal.*

Figure 10 shows the X-ray spectrum of copper that was analysed with a LiF monocrystal. Well-defined lines are superimposed on the continuous brems spectrum. The glancing angles of these lines remain unaltered when the anode voltage is varied. This indicates that these lines are characteristic X-ray lines. The two line pairs can be assigned to first-order and second-order interferences \((n = 1 \text{ and } n = 2)\). Table 1 shows the glancing angles \(\vartheta\) that were determined with the aid of Figure 10 and also the energy values for the \(K_a\) and \(K_b\) line of copper that were calculated with the aid of equation (4).
Task 2: Analyse the intensity of the copper X-radiation as a function of the Bragg angle and with the aid of a KBr monocrystal.

If the LiF monocrystal is replaced by the KBr monocrystal (Fig. 11), interferences up to the fourth order can be observed due to the larger interplanar spacing of the crystal. The spectrum of the bremsstrahlung in Figure 11 shows a clear intensity step at $\theta = 8.0^\circ$. This corresponds to the $K$-edge absorption value of bromine ($E_K = 13.474$ keV) with $n = 1$ that can be expected in theory. The $K$-edge absorptions of potassium, lithium, and fluorine cannot be observed in this area of the bremsstrahlung spectrum, since the intensity is too low.
Task 3: Determine the energy values of the characteristic X-rays of copper and compare them with the values that were determined based on the corresponding energy-level diagram.

Table 1 shows the glancing angles $\vartheta$ that were determined with the aid of Figures 10 and 11 and also the energy values for the characteristic X-ray lines of copper that were calculated with the aid of equation (4). Based on the energy values of the characteristic lines of Tasks 1 and 2, the following mean values result: $E_{K\alpha} = 8.010$ keV and $E_{K\beta} = 8.862$ keV. A comparison with the corresponding values of (1) shows good correspondence.

<table>
<thead>
<tr>
<th>LiF crystal</th>
<th>$\vartheta$</th>
<th>Line</th>
<th>$E_{exp}$/keV</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\vartheta$=1</td>
<td>22.7</td>
<td>$K\alpha$</td>
<td>7.974</td>
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<tr>
<td></td>
<td>20.4</td>
<td>$K\beta$</td>
<td>8.830</td>
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<tr>
<td>$\vartheta$=2</td>
<td>50.3</td>
<td>$K\alpha$</td>
<td>8.005</td>
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<tr>
<td></td>
<td>44.0</td>
<td>$K\beta$</td>
<td>8.857</td>
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</table>

<table>
<thead>
<tr>
<th>KBr crystal</th>
<th>$\vartheta$</th>
<th>Line</th>
<th>$E_{exp}$/keV</th>
</tr>
</thead>
<tbody>
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<td>$\vartheta$=1</td>
<td>13.6</td>
<td>$K\alpha$</td>
<td>8.018</td>
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<tr>
<td></td>
<td>12.3</td>
<td>$K\beta$</td>
<td>8.831</td>
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<td>$\vartheta$=2</td>
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<td></td>
<td>57.7</td>
<td>$K\beta$</td>
<td>8.919</td>
</tr>
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</table>

Note

The evaluation of the two spectra can be varied as follows: Use the energy values of the characteristic lines that were determined for one of the spectra in order to determine the interplanar spacing of the analyser crystal that was used for the other spectrum.
“measure” software

With the “measure” software, the peaks in the spectrum can be determined rather easily:

- Click the button $\mathbb{Q}$ and select the area for the peak determination.
- Click the button $\mathbb{Q}$ “Peak analysis”.
- The window “Peak analysis” appears (see Fig. 12).
- Then, click “Calculate”.
- If not all of the desired peaks (or too many of them) are calculated, readjust the error tolerance accordingly.
- Select “Visualise results” in order to display the peak data directly in the spectrum.

![Image of peak analysis software]

**Fig. 12**: Automatic peak analysis with “measure”