

X-ray Diffraction

Principle

The object of this experiment is to investigate some of the uses of X-ray diffraction and to understand something of the generation, diffraction and detection of X-rays.

There are three parts to this experiment.

1. To measure the characteristic X-ray spectra of copper.
2. To measure the intensity of these X-rays as a function of anode current and voltage.
3. To determine Planck's constant from the onset of the Bremsstrahlung radiation as a function of anode voltage.

Theory

When electrons of high energy strike the metallic anode of an X-ray tube, X-rays with a continuous energy distribution (the so-called bremsstrahlung) are produced. These are caused by the rapid deceleration of the electrons as they hit the anode. The energies of these X-rays will be dependent on the energy, and hence the anode voltage, of the initial electrons.

In addition to the bremsstrahlung there are 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, superimposed on the continuum. They are produced as the impact of an electron on an anode atom 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 appears as an X-ray which is specific for the anode atom. Figure 1 shows the energy level scheme of a copper atom. Characteristic X-rays produced from either the $L \rightarrow K$ or the $M \rightarrow K$ transitions are called K_α and K_β lines respectively. $M_1 \rightarrow K$ and $L_1 \rightarrow K$ transitions do not take place due to quantum mechanical selection rules.

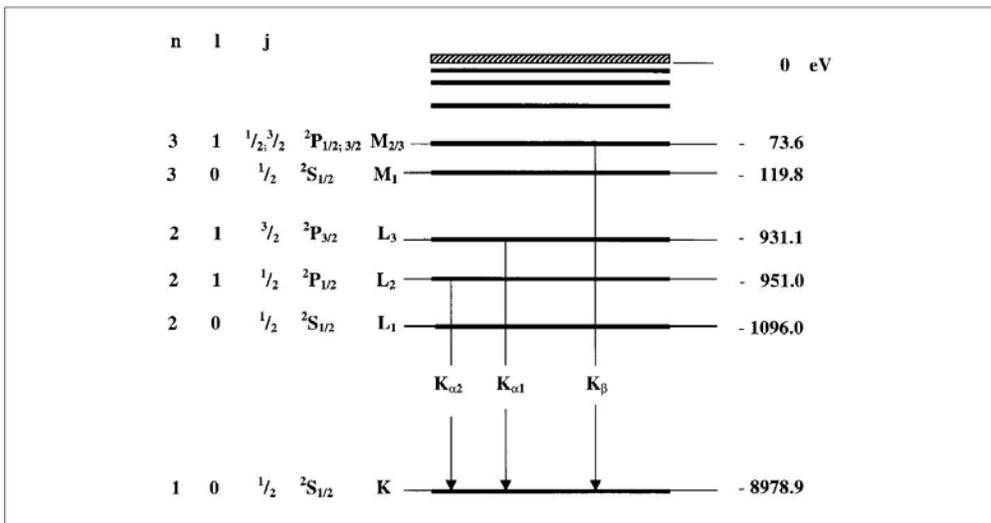


Figure 1. The energy levels of copper ($Z = 29$). The atomic energy values were taken from the "Handbook of Chemistry and Physics", CRC Press Inc., Florida.

From figure 1 it can be seen that characteristic lines for copper with the following energies should be obtained,

$$E_{K\alpha} = E_K - \frac{1}{2}(E_{L2} + E_{L3}) = 8.038keV \quad (1)$$

$$E_{K\beta} = E_K - E_{M2/3} = 8.905keV \quad (2)$$

$E_{K\alpha}$ is used as the mean value of the lines $K_{\alpha1}$ and $K_{\alpha2}$.

The analysis of polychromatic X-rays is made possible due to the diffraction of the X-rays by a crystal. When X-rays of wavelength, λ , strike a crystal at a glancing angle θ , constructive interference after scattering only occurs when the path difference Δ of the partial waves reflected from the lattice planes is one or more wavelengths (Fig. 2).

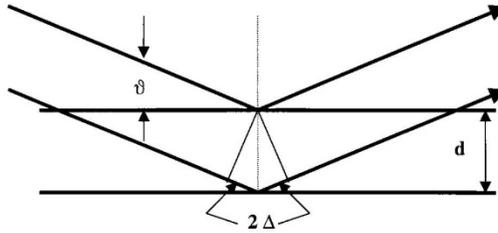


Figure 2. Bragg scattering from lattice planes

Constructive interference occurs when the difference in path length of the two beam is equal to a whole number of wavelengths. This situation is called the Bragg equation,

$$2d \sin \theta = n\lambda \quad (3)$$

Where d is the inter-plane spacing of the crystal and n is the order of diffraction. If d is known, then the energy of the X-rays can be calculated from the glancing angle θ , which is obtainable from the spectrum, and by using the relationship,

$$E = hf = hc / \lambda \quad (4)$$

On combining (3) and (4) we obtain:

$$E = \frac{nhc}{2d \sin \theta} \quad (5)$$

Planck's constant	$h = 6.6256 \times 10^{-34} \text{ Js}$
Velocity of light	$c = 2.9979 \times 10^8 \text{ m/s}$
Lattice constant of LiF (100)	$d = 2.014 \times 10^{-10} \text{ m}$
	$1 \text{ eV} = 1.6021 \times 10^{-19} \text{ J}$

Part 1. Measure the Characteristic X-ray spectra of Copper

1. The intensity of the X-rays emitted by the copper anode at maximum anode voltage and anode current is to be recorded as a function of the Bragg angle, using an LiF crystal as analyser.
2. The energy values of the characteristic copper lines are to be calculated and compared with the energy differences of the copper energy levels.

Set-up and procedure

For these experiments you will use the Phywe X-ray diffractometer. The equipment should be set up for you before you start the experiment. It has two means of control. Manual (via the front panel) and PC based. You will use both during this experiment. In manual mode you select the function you wish to change using the buttons on the front panel. Change the value using the dial and press “Enter” to set the value. *After the diffractometer has been switched to control by the computer it needs to be switched off and on to return it to manual mode.*

NOTE: This equipment is NOT a toy. X-rays can be very dangerous and the equipment is built with interlocks to stop the X-ray source being turned on when the door is open. Do not place anything inside the machine unless you have been told to do so.

If you have ANY problems or questions ask one of the demonstrators.



Figure 1. The x-ray diffractometer to be used in this experiment

For this part of the experiment you will use the PC to control the diffractometer. The following settings are recommended for the recording of the spectra:

- 2:1 coupled mode

- Integration time 2 s; angle increment 0.1°
- Scanning range 3° - 53° using the LiF crystal,
- Anode voltage $V_A = 35$ kV; anode (emission) current $I_A = 1$ mA
- 1mm aperture (Ask the demonstrators for assistance in changing this)

When you have recorded the spectra make sure that you save the data as a text file. You should obtain a spectrum similar to that shown in figure 4. You will see that there are well-defined lines superimposed on the bremsstrahlung continuum. The angles at which these lines are positioned remains unaltered on varying the anode voltage. This indicates that these lines are the characteristic copper lines. The first pair of lines belongs to the first order of diffraction, $n = 1$, whilst the second pair belongs to $n = 2$. Use the values for the Bragg angle from your spectra to determine the energies of the K_α and K_β lines for both diffraction orders. Compare the values with the theoretical values expected from figure 1.

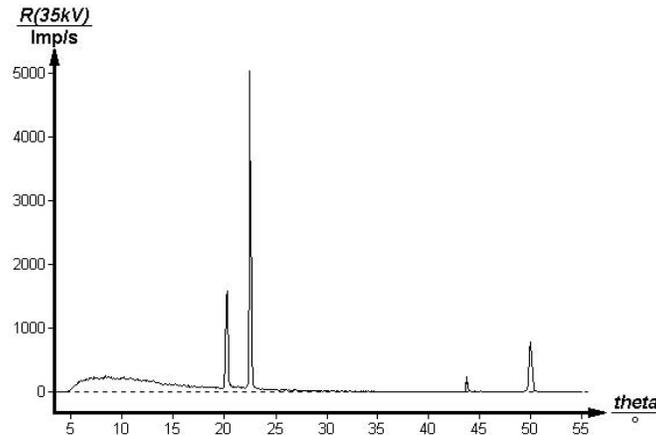


Figure 4. X-ray intensity of copper as a function of the glancing angle; LiF (100) crystal as Bragg analyzer

Part 2. Measure the intensity of the characteristic lines as a function of anode current and voltage

1. The intensities of the characteristic K_α and K_β radiations are to be determined as a function of both the anode current and the anode voltage, and be plotted graphically.
2. The results of the measurement should be compared with the theoretical intensity formula.

Set-up and procedure

For this part of the experiment you will use the diffractometer in manual mode. You will need to move to the peak of the $n=1$ K_α and K_β lines, determined in part 1, and record their intensity as a function of anode voltage and current.

Note: When moving the diffraction angle of the goniometer make sure that it is in coupled mode. (i.e. for an angle θ that the crystal moves through, the detector moves through an angle of 2θ .) Look at figure 2 to see why this should be so.

The following settings are recommended for the recording of the spectra:

- Coupled mode
- Gate time 10 s (This is equivalent to the integration time in the software)
- $V_A = 35$ kV = constant; $I_A = 1$ mA...0.1 mA in steps of 0.1 mA
- $I_A = 1$ mA = constant; $V_A = 35$ kV...11 kV in steps of 3 kV
- 2mm aperture

The gate time is the time the detector counts for. Increasing it to 10s reduces the noise in the measurements

Note: Never expose the counter tube to primary radiation for a long length of time.

Data analysis

In the X-ray source the electrons which are emitted from the cathode and accelerated towards the anode are capable of ionizing the atoms of the anode material. When this occurs in the K shell, the resulting vacancy can be filled, for example, by electrons from the L or M shell. The change in energy of this electron is emitted as so-called characteristic K_α and K_β X-rays. The intensity (measured pulse rate) of the characteristic K radiation can be approximated by,

$$N = B.I_A.(V_A - V_K)^{3/2} \quad (6)$$

where B is a constant; I_A = the anode current; V_A = the anode voltage and V_K = the ionization potential of the K level.

Plot the pulse rate, N , as a function of the anode current. You may see that at high pulse rates the results fall below the linear behaviour expected from equation 6. This is due to the dead time of the detector (After recording a pulse it takes a while to recover before it can record another. This can be corrected for by using equation 7 to remove the effect of the dead time from your data. Where N is the true pulse rate, N_0 is the measured pulse rate and τ is the dead time ($\tau \approx 50$ μ s for the counter tubes we use).

$$N = \frac{N_0}{1 - \tau.N_0} \quad (7)$$

Plot all of your data to show that equation 6 is correct.

Part 3 Determine Planck's constant from the onset of the Bremsstrahlung radiation as a function of anode voltage.

1. The intensity of the Bremsstrahlung X-rays emitted by the copper anode at various anode voltages (in 2kV steps from 35kV) are to be recorded as a function of the Bragg angle.
2. The short wavelength limit (= maximum energy) of the Bremsstrahlung is to be determined for the spectra obtained in (1).
3. The results are to be used to determine Planck's constant

Set-up and procedure

For this part of the experiment you will use the PC to control the diffractometer. The following settings are recommended for the recording of the spectra:

- 2:1 coupled mode
- Integration time 2 s; angle increment 0.1°
- Scanning range 3° - 15° using the LiF crystal,
- Anode voltage $V_A = 35$ kV; anode current $I_A = 1$ mA
- 2mm Aperture

Record and save the spectrum at each anode voltage and determine the wavelength at which the onset of the bremsstrahlung occurs. You should use these values of the onset wavelength to determine Planck's constant

Data Analysis

The positive voltage V_A on the anode of the X-ray tube causes the electrons, which are emitted from the cathode with a low (thermal) energy distribution, to accelerate. On reaching the anode, the electrons have the kinetic energy

$$E_{Kin} = eV_A \tag{8}$$

A portion of the electrons will be progressively slowed down on arrival, and their kinetic energy will be converted to electromagnetic radiation with a continuous energy distribution. Some electrons, however, will have their kinetic energy converted into electromagnetic radiation in a single step, so giving the bremsstrahlung a short wavelength limit (see figure 5). This relationship can easily be derived from Einstein's energy equation, according to which:

$$E_{Kin} = eV_A = h \cdot f_{Max} = h \frac{c}{\lambda_{Min}} \tag{9}$$

Where h = Planck's constant, c = velocity of light = 2.9979×10^8 m/s, e = elementary charge = 1.6021×10^{-19} C

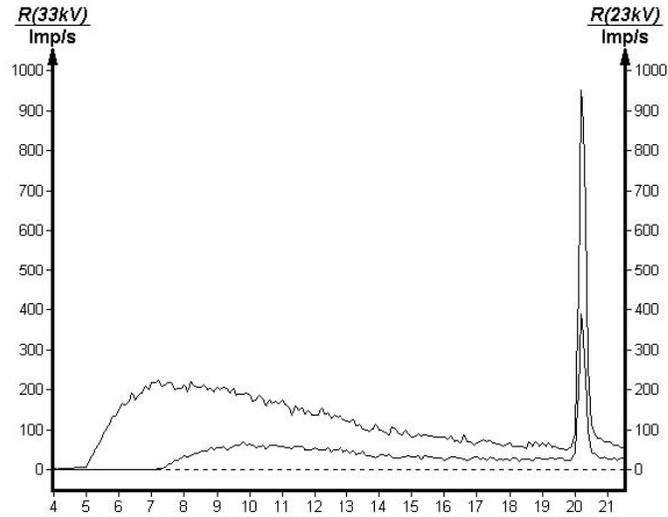


Figure 5. Bremsstrahlung as function of two anode voltages. The x-axis is the crystal angle θ in degrees.

Substituting equation 3 into equation 9 gives,

$$\frac{1}{V_A} = \frac{2ed \sin \theta_{Min}}{hc} \quad (10)$$

From your data determine θ_{Min} for the Bremsstrahlung at each voltage and hence draw a graph to determine Planck's constant.