The utility of X-ray diffraction in the study of X-rays

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ABSTRACT

The properties of X-rays are investigated using the technique of X-ray diffraction. X-rays produced from a copper X-ray tube are diffracted by a LiF crystal to reproduce the characteristic spectra of Copper and study the diffracted X-rays. Characteristic peaks are found to have energies $E_{K_\alpha} = 7.98 \pm 0.04$ keV and $E_{K_\beta} = 8.83 \pm 0.05$ keV for the $n = 1$ order of diffraction, in agreement with reference values. An approximation for X-ray intensity as a function of anode voltage and current is verified up to the limits of the apparatus and may provide use in the calibration of copper-anode diffractometers. Study of the onset of Bremsstrahlung radiation provides a method of experimentally determining Planck's constant, found to be $h = 5.80 \pm 0.50 \times 10^{-34}$, which agrees with the reference value – the main cause of the discrepancy being attributed to detector noise. Therefore, the utility of X-ray diffraction in the study of X-rays is demonstrated.

1 INTRODUCTION

X-ray diffraction is commonly used to investigate the structures of crystals however, it may also be used to investigate the properties of X-rays, as this paper will demonstrate.

A diffractometer produces X-rays with an X-ray tube by accelerating electrons onto a metal anode where collisions with anode atoms give off a spectrum of X-rays, known as Bremsstrahlung radiation, due to rapid deceleration. X-rays, of wavelength λ, may then be diffracted by a crystal with lattice spacing d, at a glancing angle θ, and constructive interference is observed by a detector when [1]

$$n\lambda = 2d \sin\theta \tag{1}$$

where n is the order of diffraction. The kinetic energy of an electron striking the anode is proportional to the anode voltage, V_A, that accelerates it [1]

$$E = \frac{hc}{\lambda} = eV_A \tag{2}$$

where e is the elementary charge, h is Planck's constant and c is the speed of light in vacuum. Electrons stopped by a single impact emit X-rays of maximum energy at a minimum angle θ_{min}, which corresponds to the onset of Bremsstrahlung radiation. Electrons with sufficient energy can ionise anode atoms resulting in electron transitions from the L and M shells to de-excite the atom, producing X-rays lines K_α and K_β, which are detected as characteristic peaks in intensity N, approximately related to the anode current I_A and anode voltage by [1]

$$N = BI_A(V_A - V_K)^{1.5} \tag{3}$$

where V_K ($= 8978.9$ eV for copper [2]) is the ionisation potential of the K level and B is a constant. By combining Eq.s (1) and (2), the energy of a peak may be found by

$$E = \frac{nhc}{2d\sin\theta}. \tag{4}$$

Furthermore, by consideration of the value θ_{min}, Eq.s (1) and (2) also give

$$\frac{1}{V_A} = \frac{2ed\sin\theta_{min}}{hc} \tag{5}$$

which provides a way to experimentally determine a value for h.

In this paper, the characteristic spectra of copper is measured to calculate the energies of the K_α and K_β lines, verify Eq. (3) and determine a value for Planck's constant using Eq. (5).

2 METHOD

A PHYWE XR 4.0 diffractometer with a copper X-ray tube and a LiF crystal ($d = 2.014$ Å [3]), capable of voltage and current ranges of 5.0 – 35.0 kV and 0.0 – 1.0 mA, was used to measure the X-ray spectra of copper. The X-rays were collimated by a 1 mm diaphragm tube to measure a spectrum over approximately $50°$ of crystal rotation in intervals of $0.1°$, and by a 2 mm diaphragm tube to measure smaller spectra with less beam divergence. Maximum anode current and voltage were used to obtain the spectrum as this produced more intense peaks that were clearly visible over detector noise, and the data was aggregated by the software, Measure.

Eq.(3) was tested using the first peak in the spectra ($\theta = 20.4°$, see figure 1) and alternately varying the anode current or voltage, randomly sampling intensities from the XR 4.0's display, which updated every 10 s. The detectors minimum time separation between measurements, known as the dead time ($\tau \approx 50\mu$s), was accounted for using the nonparalyzable model [4], giving true intensities

$$N = \frac{N_0}{1 - \tau N_0} \tag{6}$$

where N_0 is the measured intensity.

For an anode voltage $V_A \approx 30$ kV, θ_{min} is predicted to occur at $\theta_{min} \approx 6°$ using Eq. (2). As a result, measurements were taken from $3 - 15°$ in intervals of $0.1°$ to investigate the onset of Bremsstrahlung radiation at 1.0 mA and varying anode voltage.

The XR 4.0 has an interlocking door with an electronic lock which must be closed for the X-ray tube to operate. The door was secured and checked before each measurement to minimise the possibility of exposure to X-rays.

3 RESULTS AND DISCUSSION

The characteristic spectra of copper is shown in figure 1 and the peaks may be identified by order of diffraction – the first two at $\theta \approx 20°$ correspond to $n = 1$, the latter two to $n = 2$ – followed by the energy transitions, of which, the smaller angle is the more energetic K_β line. The peaks were fitted using a Gaussian model to determine the centroid position (see the inset of figure 1 for an example) and the centroid uncertainty was taken as half the width of the peak. Using Eq. (4) with reference values of h, c and d [2, 3], the transition energies were determined as shown in table 1, where the errors on the centroid positions were propagated using the general formula for error propagation [5]. The values agree with

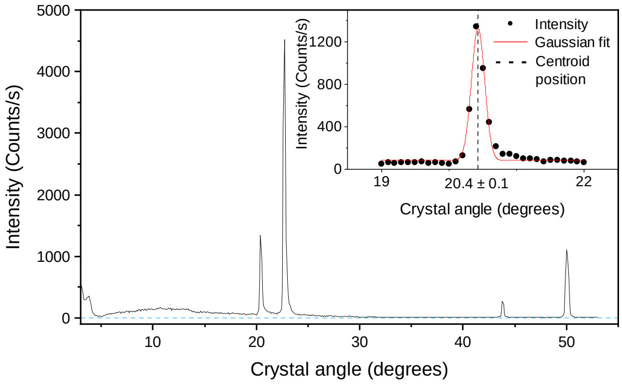

Figure 1. Characteristic X-ray spectra of copper. Inset shows an example Gaussian peak fit of the $n = 1$, K_β peak, with centroid position.

Table 1. Experimentally determined energy values for the K_α and K_β characteristic energy lines of copper.

Transition	n	Energy (keV)	Error (keV)
K_α	1	7.98	0.04
	2	8.04	0.02
K_β	1	8.83	0.05
	2	8.89	0.02

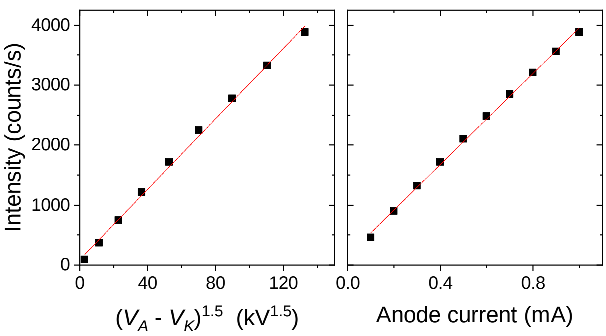

Figure 2. Dead-time-corrected intensity as a function of anode current and anode voltage based on Eq. 3, with linear fits.

the literature [2] to within three standard deviations, demonstrating the resolving power of X-ray diffraction.

Figure 2 shows the dead-time-corrected intensity as a function of anode voltage and anode current. Linear fitting models were applied resulting in correlation coefficients of approximately 1 for both plots, indicating very good linear fits [5]. Standard errors were calculated for each sampling of intensity and propagated to Eq. (6) [5]; instrumental errors for anode current and voltage were taken as half the resolution. Contributions from both sources of error were small ($\approx 1\%$). The rate of increase of intensity decreases slightly at higher voltages and currents suggesting that Eq. (3) may not be a valid approximation beyond the range investigated, as is suggested by the work of Worthington *et al* [6], because the intensity saturates. The constant of proportionality was calculated using the gradients of figure 2 to be $B = 0.95 \pm 0.1\mathrm{s}^{-1}\mathrm{A}^{-1}\mathrm{eV}^{-1.5}$ and may be useful for calibrating copper-tube diffractometers of similar operating voltages.

Values for θ_{min} were obtained by identifying the crystal angle corresponding to the minimum intensity in the predicted region ($\pm 0.5°$). This ad hoc method was chosen because a significant level of noise was detected, making the onset of Bremsstrahlung radiation difficult to identify so, it was assumed that measurements before the minimum intensity were purely noise. Figure 3 shows the results with two linear fits – one excluding the outlier – and their gradients. The fit excluding the outlier provides a value of Planck's constant as $h = 5.80 \pm 0.50 \times 10^{-34}$ Js and shows better agreement to the literature [2] than the alternative fit. The discrepancy is most likely due to inaccuracies in identifying θ_{min} which may be improved by reducing detector noise, possibly by cooling the diffractometer to lessen any thermal noise.

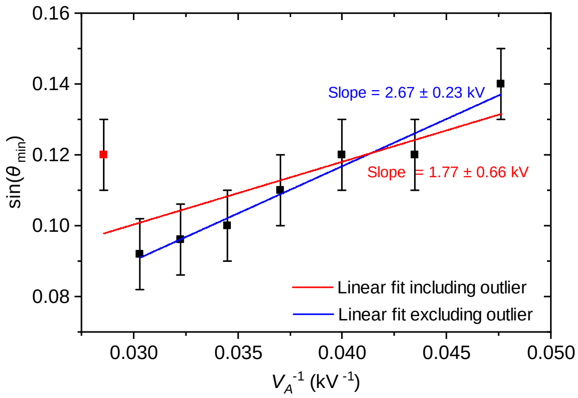

Figure 3. Reciprocal anode voltage dependence on the minimum crystal angle with two linear fits (the red fit includes the red outlier), and the respective slopes.

4 CONCLUSIONS

X-ray diffraction has been used to reproduce the characteristic spectra of copper and calculate the transition energies $E_{K_\alpha} = 7.98 \pm 0.04$ keV and $E_{K_\beta} = 8.83 \pm 0.05$ keV for the $n = 1$ order of diffraction, which agree with the literature. An approximation for X-ray intensity as a function of anode voltage and current has been verified for the ranges $0.1 - 1.0$ mA and $11.0 - 35$ kV, with the constant of proportionality $B = 0.95 \pm 0.1\mathrm{s}^{-1}\mathrm{A}^{-1}\mathrm{eV}^{-1.5}$. This result may be useful for the calibration of copper-tube diffractometers of similar anode voltages but is expected to breakdown at higher values due to intensity saturation. Planck's constant was determined to be $h = 5.80 \pm 0.50 \times 10^{-34}$ Js by studying the onset of Bremsstrahlung radiation and agrees with the reference value but, should be made more accurate by reducing detector noise. These results demonstrate the utility of X-ray diffraction in the study of X-rays.

References

[1] – B.D.Cullity, S.R.Stock, Elements of X-Ray Diffraction, Third Edition, Pearson Education, Sections 1.3-1.4 (2013).

[2] – David R. Lide, CRC Handbook of Chemistry and Physics, CRC Press, Section 1 pg. 1-2 & Section 10 pg.196 (2005).

[3] – Charles Kittel, Introduction to Solid State Physics Eighth edition, John Wiley & sons, pg. 66 (2005).

[4] – Glenn F. Knoll, Radiation Detection and Measurement Third edition, John Wiley & Sons, pg. 120 (2000).

[5] – John R. Taylor, An Introduction to Error Analysis Second edition, University Science Books, Sections 5.5 - 5.7, 9.3 (1997).

[6] – C R Worthington and S G Tomlin (1956), Proc. Phys. Soc. A **69** 401, DOI: https://doi.org/10.1088/0370-1298/69/5/305