King Fahd University of Petroleum & Minerals

Physics Department

PHYS-403: Experimental Physics-II

XRD: A Report on X-ray Diffraction Experiment

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To

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Contents

1 INTRODUCTION

1.1 Objectives

For **part.i** of the experiment we aim to achieve the following objectives:
1. Investigating the fine structure of the characterstic x-radiation of molybdenum by means of Bragg reflection at an NaCl monocrystal in the fifth diffraction.

2. Identifying the characterstic K_α, K_β and K_γ lines.

3. Resolving the fine structure of the K_α line as a line doublet and determining the wavelength interval $\Delta\lambda$ within the doublet.

While for **part.ii** of the experiment we aim to achieve the following objectives:
1. To determine the limit wavelength λ_{min} of the bremsstrahlung countlnuum as a function of the high voltage U of the x-ray tube.

2. To confirm the Duane-Hunt relation.

3. To determine Planck's constant

Finally for **part.iii** of the experiment we aim to achieve the following objectives:
1. To investigate Bragg reflection at an NaCl monocrystal using the characteristic x-ray radiation of molybdenum.

2. To determine the wavelength of the characterstic $K_\alpha and K_\beta$ x-ray radiation of molybdenum.

3. To confirm Bragg's law of reflection

4. To verify the wave nature of x-rays.

1.2 Theory of part.i

The theory behind investigating the fine structure of the characteristic x-radiation of molybdenum by means of Bragg reflection at an NaCl monocrystal in the fifth diffraction is based on the phenomenon known as Bragg's law. When X-rays are incident on a crystalline material at precise angles, some of this radiation can be reflected due to constructive interference. The angle at which this reflection occurs (called the Bragg angle) is dependent on properties of the crystal lattice such as spacing between atoms and atomic scattering factors. By measuring the position and intensity of the reflections, it is possible to deduce information about the inner structure of materials such as molybdenum. In this case, using a NaCl crystal with a fifth order diffraction, it should be possible to obtain more detailed information about other elements present in combination with molybdenum. Such phenomenon ease the way to identify the K-lines and resolve the fine structure of the K_α lines as a line doublet.

1.3 Theory of part.ii

The limit wavelength of the bremsstrahlung continuum in XRD is determined by the voltage of the x-ray tube. As the voltage increases, the cutoff wavelength will decrease, i.e. shorter wavelengths will be generated. The maximum cutoff wavelength can be calculated as U / (2Z) where Z is the atomic number of the target material. Now, it has been found that there is a promotional relationship between the limit wavelength and the tube high-voltage. This relation is called Duane-Hunt relationship, which is a fundamental relationship between the wavelength and frequency of electromagnetic radiation, also known as the 'inverse linear relationship'. This simply states that as the wavelength increases, the frequency decreases and vice versa. This has become an important concept in physics, especially for the study of the visible light spectrum and related mechanisms. Now, this relationship can ease the way to calculate Plank's constant. Importantly, Planck's constant cannot be directly determined by XRD. Instead, Planck's constant and other constants of nature can be inferred indirectly by measuring the physical properties (such as crystal structure and unit cell parameters) of a material using X-ray diffraction method. These measurements can then be used to calculate and estimate the values of constants such as Planck's constant, which is a fundamental value that helps characterize all matter in the universe, since it determines the quantized energy of molecules, atoms, and photons and that we will try to do here.

1.4 Theory of part.iii

Bragg's law states that when x-rays are diffracted by a single crystal at an angle, reflections occur when the product of the interplanar spacing d and the sine of the diffraction angle θ is equal to an integer multiple of the wavelength λ of the x-ray. This allows for direct determination of atomic spacing in a crystal, as well as creation of detailed structure maps for crystalline materials. The Bragg reflection process makes use of constructive interference when two waves meet and can be used to study a wide range of stimulii, for example acoustic, optical and X-ray energies. Diffraction from a single crystal enables analysis from both momentum transfer (associated with the scattering angle) and energy transfer (associated with the diffractometer spectrometer setting). Thus it is possible to measure channels in momentum space that are inaccessible by other methods. By combining Bragg reflections and diffractions it is possible to create high resolution structure maps that are crucial in many scientific disciplines such as drug discovery, materials science and biochemistry.

2 EXPERIMENTAL SET-UP

For all the three parts the set up is identical (as shown in Figure.1) except that the parameters are different.

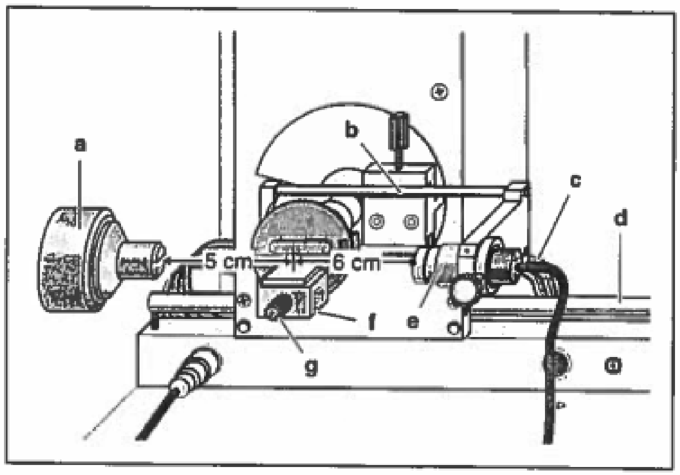

Figure 1: XRD set up

2.1 PROCEDURE

1. prepare your sample
2. Put it on the holder carefully
3. locate the detector correctly according to the part you are dealing with
4. close the XRD door
5. Turn-on the XRD
6. Open the allocated software for the XRD device
7. set up the required parameters
8. scan and obtain your results

For part.i:
U = 35 kV , I = 1.00 mA , $\Delta\beta = 0.1^o$
for 1st order of diffraction:
$\theta_i = 5.5^o$ and $\theta_f = 8.0^o$, $\Delta t = 10s$
for 5th order of diffraction:
$\theta_i = 32.5^o$ and $\theta_f = 40.5^o$, $\Delta t = 400s$

For part.ii:
we were changing the voltages as:
U = 22, 24, 26, 28, 30, 32, 34, 35 (kV)
while current was fixed to I = 1.00 mA
and Δt = 30, 30, 20, 20, 10, 10, 10, 10 (s) corresponding to each voltage, respectively. Finally the able β is around 6^o while change in angle was fixed to be $\Delta\beta = 0.1^o$

For part.iii:
U = 35 kv, I = 1.00 mA , $\Delta t = 10s$ and $\Delta\beta = 0.1^o$
while $\theta_i = 2^o$ and $\theta_f = 25^o$.

3 RESULTS & DISCUSSION

3.1 Part.i

part a) first diffraction:

Lines	Measured $\frac{\lambda}{pm}$	Calculated $\frac{\lambda}{pm}$	error %
K_α	73	71.08	2.7
$K_{\beta+\gamma}$	65	63.09	3

Figure 2: Diffraction spectrum of x-rays in Brag reflection in the first order at an NaCl monocrystal. Left peak refers to K-beta while right peak refers to K-alpha

part b) fifth diffraction:

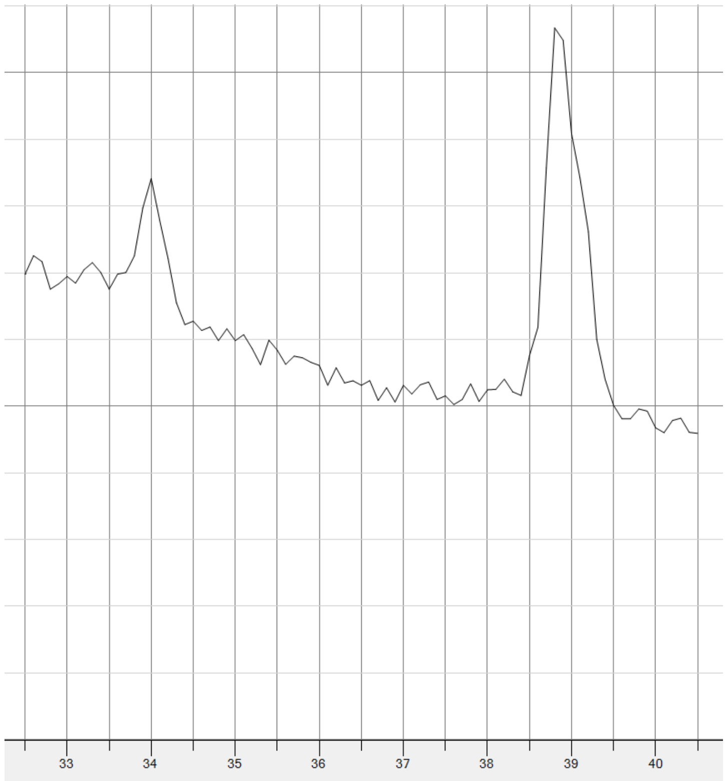

Figure 3: Diffraction spectrum of x-rays in Brag reflection in the fifth order at an NaCl monocrystal. peaks corrosponds to K-gamma, K-beta, K-alpha1 & K-alpha2 from the left to the right respectively

Lines	Measured $\frac{5.\lambda}{pm}$	$\frac{\lambda}{pm}$	Calculated $\frac{\lambda}{pm}$	error %
$K_{\alpha 1}$	358	71.6	70.93	1
$K_{\alpha 2}$	359.2	71.9	71.36	0.8
K_{β}	313.2	62.6	63.26	1
K_{γ}	311	62.2	62.09	0.2

Results

The characteristic K-alpha and K-beta lines that have been observed in the 1st diffraction order split into doublets. Which can be observed in the 5th diffraction.

The fine structure of the K-alpha doublet is a consequence of the fine structure of the L-shell. The K-beta doublet is composed of the pure K-beta line and the K-gamma line.

3.2 Part.ii

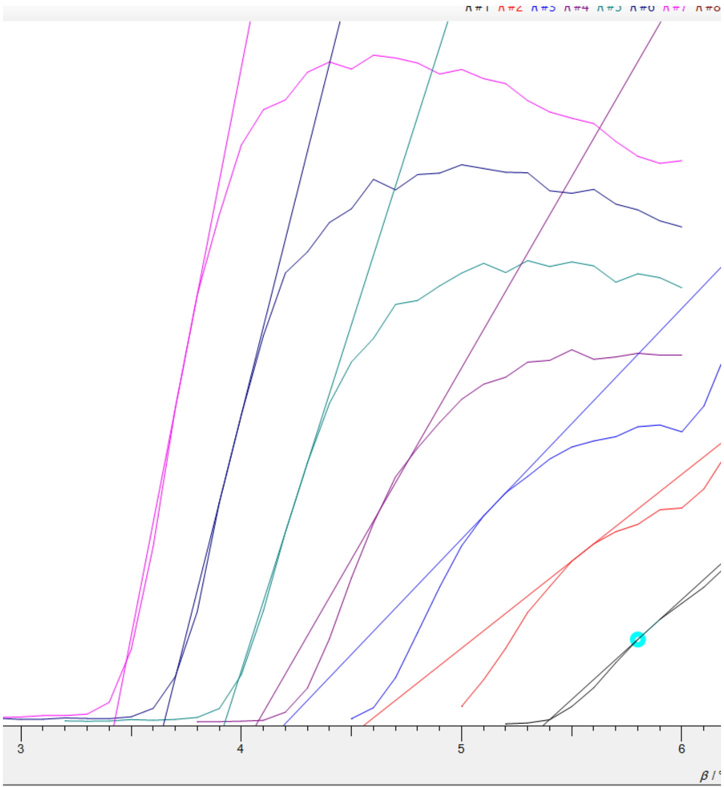

Figure 4: Sections from the fdiffraction spectra of x-radiation for the tube high voltage U values

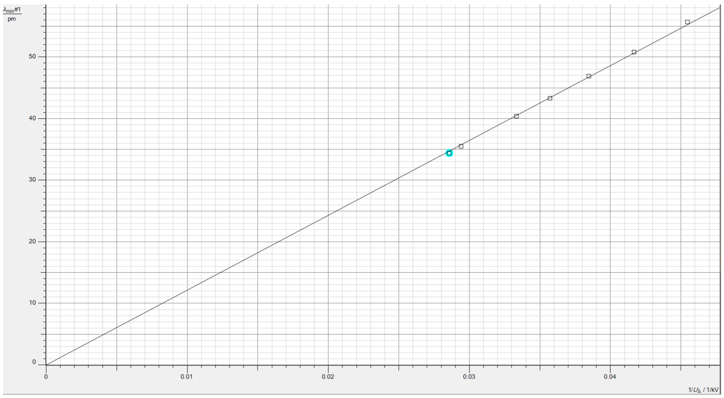

Figure 5: Evaluation of the data for confirmu=ing the Duane-Hunt relation and determining Planck constant

The best-fit straight line gives us:
A = 1223 pm . kV

Now using the following equation : $E_{max} = h.\nu_{max}$ we can get Planck's constant:

$$h_{measured} = 6.54 \cdot 10^{-34} J \cdot s$$
while
$$h_{calculated} = 6.626 \cdot 10^{-34} J \cdot s$$
so
error = 1.3 %

3.3 Part.iii

n	θ_{K_α}	$\frac{\lambda(K_\alpha)}{pm}$
1	7.36^o	71.35
2	15.92^o	71.38
3	21.41^o	71.29

Table 1: Measured glancing angles of the Mo K-alpha line & calculated λ for the first three orders of diffraction

n	θ_{K_β}	$\frac{\lambda(K_\beta)}{pm}$
1	6.49^o	62.89
2	12.39^o	62.98
3	18.96^o	62.83

Table 2: Measured glancing angles of the Mo K-beta line & calculated λ for the first three orders of diffraction

	$\frac{\lambda(K_\alpha)}{pm}$	$\frac{\lambda(K_\beta)}{pm}$
Mean value	71.34	62.9
Literature value	71.08	63.09
% error	0.4	0.3

Table 3: Mean values for the characteristic wavelength λ

Results

The results that have been found assures that Bragg's law is valid in addition to the confirmation of the x-rays wave nature.

4 CONCLUSION

In conclusion, XRD was insightful in terms of matching the theory and experiment. Reproducing a fundamental constant such as Planck constant alone is a pleasure. However, XRD analysis is an incredibly useful tool for studying material properties, such as crystallinity and phase identification. Its non-destructive methods make it particularly useful in many applications, allowing scientists to study the dynamic characteristics of a sample without damaging it. It is a cost-effective way to gain insight into the structure and composition of materials. Overall, XRD has demonstrated its utility in analyzing samples across numerous industries with results that are reliable and reproducible.

5 REFERANCES

[1] LAB MANUAL