X-Ray Diffraction

Joshua Liu
Shivani Vinayak Hegde
20674109
Mayar Mohamed
20699909
Department Physics and Astronomy
Waterloo University

CONTENTS

LIST OF FIGURES

LIST OF TABLES

X-Ray Diffraction

***Abstract*—X-ray diffraction has been a popular technique for decades and continues to be used across the world. A demonstration of this technique was performed to identify the identity of a metal wire, as well as a crystal powder. While the identities were not quite found in the analysis, but the miller indices of the metal sample were found to be: (39.4; 585) (46.2;623) (67.6; 451) (81.7; 624) (86.2; 200) (104.4; 126) (118.8;402).**

I. Introduction

X-Ray Diffraction (XRD) is most famous for its help in finding the double helical structure of DNA as well as its many uses in the classification of proteins, metals, and crystals. Providing atomic resolution images of the sample, it allows researchers to study the arrangement of atoms as well as bond lengths, bond angles, and material composition. As the name implies, it uses an x-ray beam focused onto the sample at an angle while a charged coupled device (CCD) on the other side detects and maps intensity peaks. The x-ray then moves to a subsequent angle and the mapping is done again. This process repeats itself about a thousand times until it has gone its specified range of motion. This intensity data is plotted either in an intensity vs the diffraction angle, which will indicate the miller indices of the diffracted crystal, or as a diffraction pattern, which would be processed using a slew of mathematical algorithms to create an image of the crystal structure. It is no wonder why this technique along with many others derived from XRD are still being used today in a wide variety of labs. In this experiment, an unknown metal wire will be examined using XRD. The output data will be in the form of an intensity vs diffraction angle plot, using this the miller indices will be identified and the identity of the metal, along with its lattice spacing should be resolved. A powder XRD will then be done with another unknown sample in a glass capillary tube. Again, the data will be plotted in an intensity vs diffraction angle plot, which will allow for the same information to be extracted.

II. Theoretical Background [1]

To get a sharp peak in the intensity of the scattered x-rays radiation, the X-rays should be reflected by the atoms in one plane, and the reflected rays from the successive planes should interfere constructively.

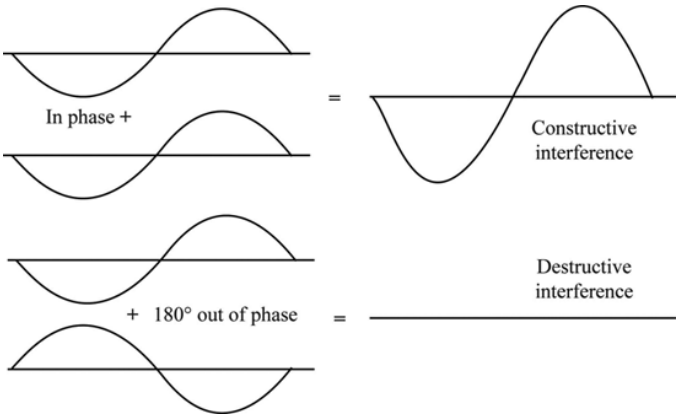

Fig. 1. Constructive interference vs destructive interference

Referring to figure 1, to get a constructive interference, the two diffracted rays should be in phase, that means that the deference between the diffracted rays from the successive planes should be equal multiple integer number of the wavelength.

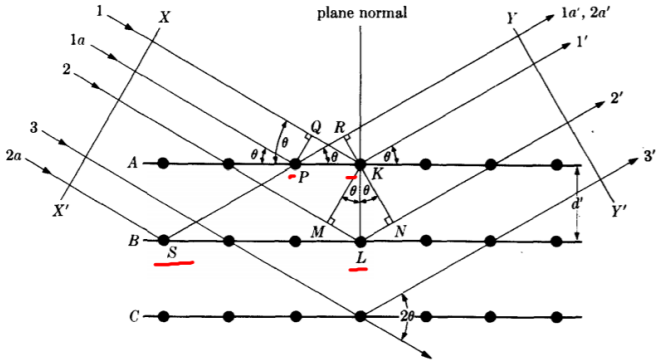

Fig. 2. Diffraction from the crystal lattice

Referring to figure 2, the difference between ray 1 and 2, is the distance **ML + LN**

$$ML + LN = d'sin\theta + d'sin\theta \tag{1}$$

the path difference is equal to a whole number n of wavelengths:

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

where n = 1,2,3..
d' is interplanar spacing
λ is the wavelength
θ is the diffraction angle
This equation is called Bragg's law and it was first introduced by W. L. Bragg. So for diffraction to occur, this

condition must be satisfied. In the experiment, we found Bragg angles θ for the sample, now it's possible to determine the crystalline structure of the sample. For cubic structure, we will assign indices to each peak position, those indices are called Miller indices, hkl. We know that the cubic crystal relation:

$$d_{hkl} = \frac{a_o}{\sqrt{h^2 + k^2 + l^2}} \tag{3}$$

In our experiment, we need to determine the lattice constant a_o for every peak in the sample.

Then we substitute Bragg's law in the above equation, we get:

$$\lambda = \frac{2a_o sin\theta}{\sqrt{h^2 + k^2 + l^2}} \tag{4}$$

Then:

$$sin^2\theta = (\frac{\lambda^2}{4a^2})(h^2 + k^2 + l^2) \tag{5}$$

Such that $(\frac{\lambda^2}{4a^2})$ is constant So for all the Bragg angles we obtained, we divide $sin^2\theta$ by set of integers to get a common quotient which is the value of $(\frac{\lambda^2}{4a^2})$. Now we divide $sin^2\theta$ by the quotient we got for each Bragg peak to get the integers of $h^2 + k^2 + l^2$.

(hkl)	$h^2+k^2+l^2$	SC	BCC	FCC
100	1	✓	×	×
110	2	✓	✓	×
111	3	✓	×	✓
200	4	✓	✓	✓
210	5	✓	×	×
211	6	✓	✓	×
220	8	✓	✓	✓
300, 221	9	✓	×	×
310	10	✓	✓	×
311	11	✓	×	✓
222	12	✓	✓	✓
320	13	✓	×	×
321	14	✓	✓	×
400	16	✓	✓	✓
410, 322	17	✓	×	×
411, 330	18	✓	✓	×
331	19	✓	×	✓
420	20	✓	✓	✓
421	21	✓	×	×

Fig. 3. Indexing Cubic Crystals

Using table 3 [2], now we can identify the Braivais lattice, whether it's primitive, Face Centered Cubic (FCC) or Body-Centered Cubic (BCC).

Finally, now we can determine our best value lattice parameter a_o

$$a_o = \frac{\lambda\sqrt{h^2 + k^2 + l^2}}{2sin\theta} \tag{6}$$

III. EXPERIMENTAL DESIGN AND PROCEDURE

A. *Description of the apparatus*

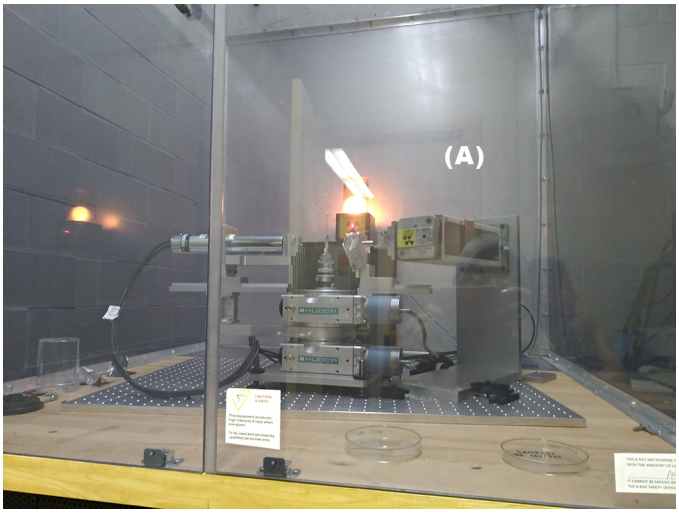

Fig. 4. apparatus components

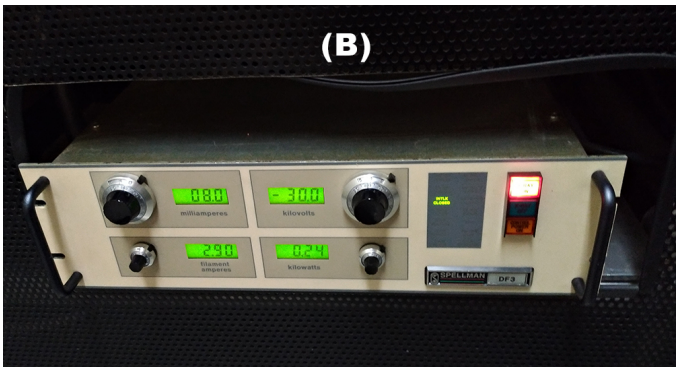

Fig. 5. apparatus components

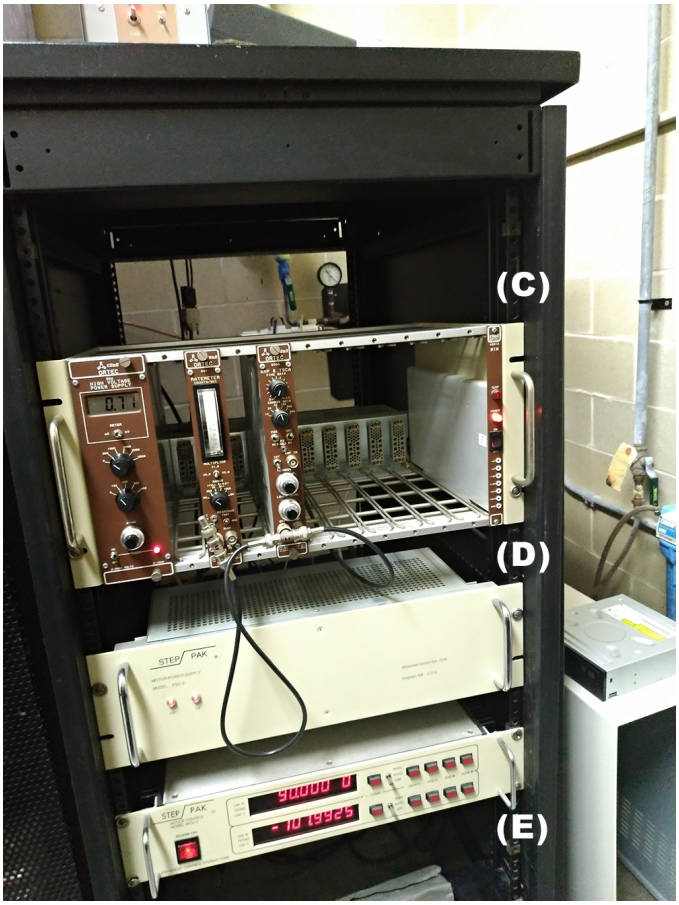

Fig. 6. apparatus components

The used apparatus consists of :

A) The experiment area, contains the detector, the source and the sample holder.
B) The filament source power supply.
C) The detector high voltage supply.
D) The stepper motor power supply.
E) Step Pak motor control model MCU-2.
F) (Not shown in the picture) The x-ray shutter control box.

B. Description of the experimental procedure

There are two parts for this experiment. For the first part, we were provided with a sample of a metallic wire in order to determine Bragg Angles so we can calculate the lattice Spacing of the metal. For the second part, we were provided with a powder sample to determine Bragg Angles, and so we can determine its crystalline structure and its lattice parameter. We started by adjusting the power supplies to the right settings as written in the provided lab manual, then we placed the sample on the goniometer.

Then we used the Lab's PC to obtain the data, the system used is Linux, we opened the terminal and used "twoc" program. We started by testing the stepper motor command, we moved the detector to a position at 10 degrees. However, we had some troubles calibrating the stepper motor to align with the program command so we had to reset the system multiple times.

After we calibrated the detector position, we were ready to start the scan. We performed the scan from 10 to 120 degrees using 1100 data points, which is 0.1° for each step. So each point was exposed to the rays for 4 seconds. As shown in figure 7, we could see the peaks while performing the experiment. We collected the data points for the two samples provided.

Fig. 7. Linux system, using 'twoc' program on terminal

IV. ANALYSIS

A. Method of Analysis and Presentation of Results

In our experiment, we chose to use the program 'twoc' through the terminal accessible in the Linux OS of the computer. This was used to perform the method of X-Ray Diffraction, as previously mentioned as XRD. As we went through the script of 'twoc', we were able to perform two experiments with 'twoc'; we measured the diffraction of the metal to possibly determine the lattice spacing of the metal and identify the unknown substances.

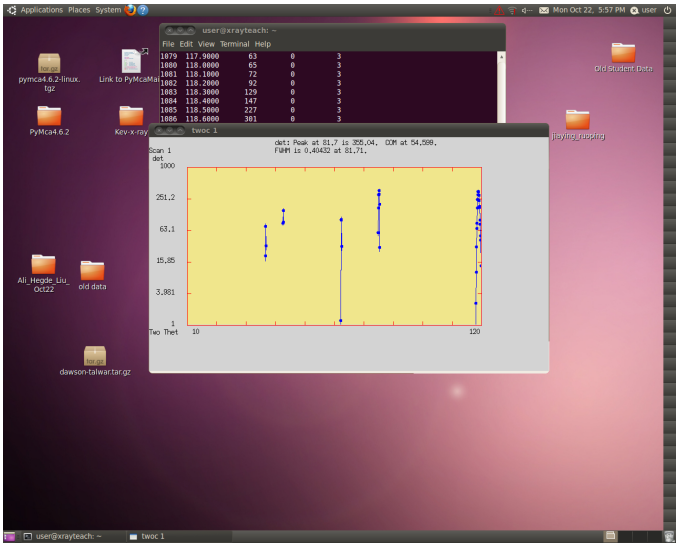

Fig. 8. Metal sample graph

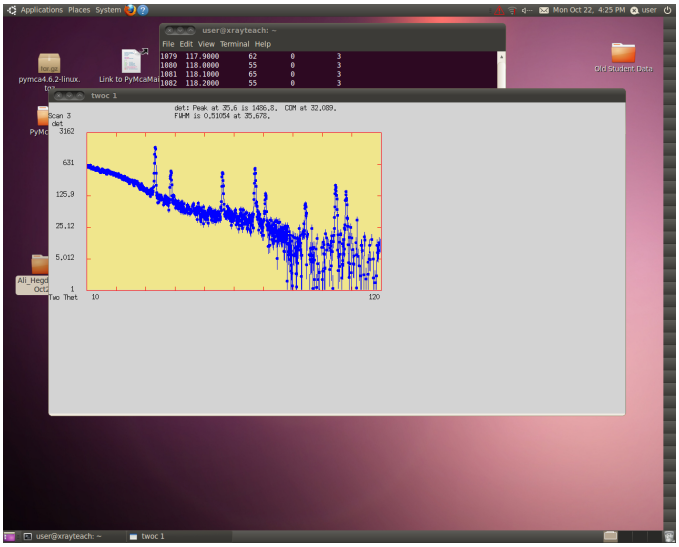

Fig. 10. Powder sample graph

Above what we see is what the program showed us was happening in the XRD, but after plotting the data using Python, we obtained the following graph.

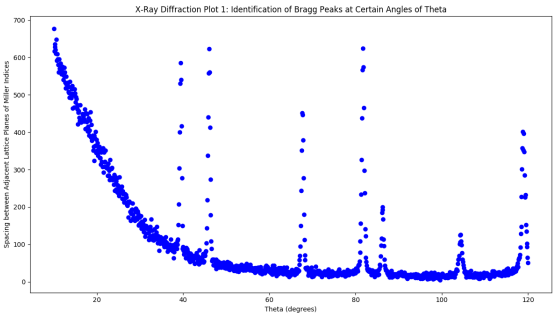

Fig. 9.

Table 10–5

Line	Intensity	$\sin^2\theta$	*hkl*
1	s	0.0462	111
2	vs	0.1198	220
3	vs	0.1615	311
4	vw	0.1790	222
5	m	0.234	400
6	m	0.275	331
7	s	0.346	422
8	m	0.391	511, 333
9	w	0.461	440
10	m	0.504	531
11	m	0.575	620
12	w	0.616	533
13	w	0.688	444
14	m	0.729	711, 551
15	vs	0.799	642
16	s	0.840	731, 553

Fig. 11. Table 10-5 from Cullity's 'Elements of X-Ray Diffraction'

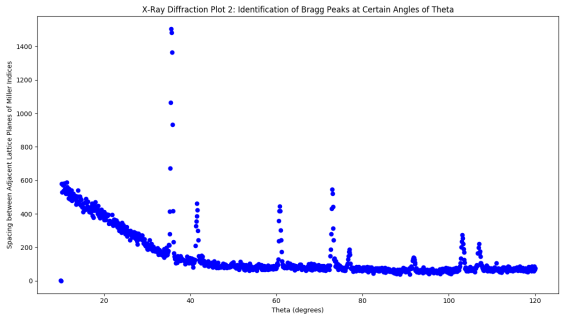

Fig. 12. Plot of Powder Sample's Spacing between Adjacent Lattice Planes of Miller Indices vs. Angles of Theta

B. Discussion of results

In Figure 9, we can see that at certain points, there are peaks that stand out from the rest of the graph. Those points are as follow: At: (Theta; Spacing between Adjacent Lattice Planes of Miller Indices for the Metal Sample); (39.4; 585) (46.2; 623) (67.6; 451) (81.7; 624) (86.2; 200) (104.4; 126) (118.8; 402)

V. CONCLUSION

In the experiment of X-Ray Diffraction, we learned about Bragg's Law, seeing how the spacing of the adjacent lattice planes of Miller indices can help us determine the lattice parameter. We were able to work with and manipulate the angles of theta to see where the Bragg peaks would appear in the process of the diffraction. It seems that the relative intensities due to the spacing depended on the $sin^2\theta$; where the greater the $sin^2\theta$, the weaker the intensity of the line, in general. The use of X-Ray Diffraction allows us to identify the most unknown crystalline compounds, this could be very helpful in identifying which sort of glass is most effective when making telescopes that require utmost precision and accuracy.

REFERENCES

[1] Cullity, B.D. (1978) Elements of X-Ray Diffraction. 2nd Edition, Addison-Wesley Publishing Company Inc., Phillippines.

[2] Saher, F. (2010). Crystal Structure Determination I. Presentation, Pakistan Institute of Engineering and Applied Sciences.

[3] Stanjek, H., Häusler, W., (2004) Basics of X-ray Diffraction, Hyperfine Interactions. 154(1):107-119 doi: 10.1023/B:HYPE.0000032028.60546.38