X-Ray Powder Diffraction

PHYS 4007

Lab Report

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ABSTRACT

This lab report focuses on X-Ray diffraction of behavior of various crystals. Copper is used as an x-ray source for the experiment. Firstly, Sodium Chloride (NaCl) crystals of miller indices (100, 110, 111) were exposed to x-rays. From which the backscatter of the NaCl Crystals was measured using a goniometer. Using this back scatter, the lattice constant 5.7601+/- 0.0051 ($\dot{A}$) of NaCl crystals was calculated. Secondly, Lithium Fluoride (LiF) sample was exposed to the X-rays and the backscatter was used to develop a photographic film. The pattern developed on the photographic film was used to analyze the wavelength spectrum of the x-rays. Finally, an unknown crystal sample was used and was exposed to x-rays for 6 hours. The setup was used to develop a photographic film. The film was analyzed to calculate the lattice constant of the sample used and thus recognize the sample. It was found that the sample used was Iron (Fe) with the lattice constant 2.866 $\dot{A}$.

THEORY

Crystal Structure and Miller Indices

In a crystal lattice the atoms are arranged in a periodic structure. The smallest repeated unit is called a unit cell. A unit cell can be of three basic forms, primitive cubic, body-centered cubic, face centered cubic. The structural difference between them is depicted in figure 1 [1] [2].

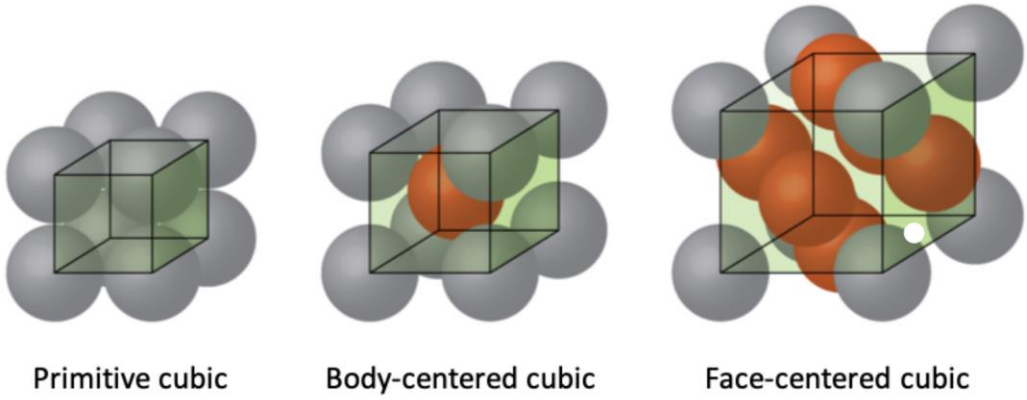

Figure 1: Various Types of Crystal Lattice structures [2]

There can be different planes of interest to study and analyze various crystal lattices. Some of these planes have been depicted in figure 2 [1].

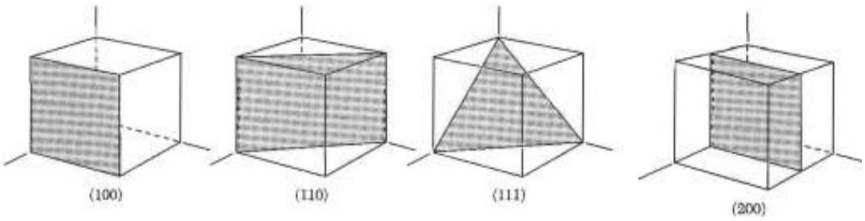

Figure 2: Different Planes to observe a cubic crystal [1]

A set of vectors can be used to define these crystal structures and planes (a_1, a_2, a_3) These vectors are defined by constant multiples of lattice vectors. As depicted in equations 1, with a being the side of the cube and X, Y, Z being the orthogonal unit vectors [1].

$$a_1 = \frac{a}{2}(-\hat{X} + \hat{Y} + \hat{Z});\ a_2 = \frac{a}{2}(\hat{X} - \hat{Y} + \hat{Z});\ a_3 = \frac{a}{2}(\hat{X} + \hat{Y} - \hat{Z}) \qquad (1a, 1b, 1c)$$

For this experiment sodium chloride (NaCl) was studied in its indices 100, 110, 111 as the magnitude of (a_1, a_2, a_3) for it is equal to the value of its lattice constant. [1]

Bragg's Law

Bragg's law defines the situation of constructive interference of waves. Such conditions are defined by equation 2. In equation 2, n defines the order of reflection, λ defines the wavelength of light, d_{hkl} defines the spacing between the planes and Θ defines the angle of interference [1].

$$n\,\lambda = 2d_{hkl}\sin\theta \qquad (2)$$

Each plane of atoms that satisfies the Braggs' law will give a bright spot and hence a peak in the spectrum results. To account for all possible results the due to various miller indices the d is calculated separately for each and then is further used to calculate the lattice constant using the miller indices, as per equation 3 [1].

$$a^2 = d_{hkl}^2(h^2 + k^2 + l^2) \qquad (3)$$

X-Ray Production

In this experiment copper was used to generate x-ray emissions. X-rays are generated by hitting the target with high energy electrons followed by the de-excitation of atom, which results in emission of x-rays. The X-rays studied in this experiment were released in three sets of emissions $K_{\alpha 1}, K_{\alpha 2}$, K_β. The first two are so close in the wavelength that they are observed as one in the spectrum and they refer to the transition from L3 and L2 shell to K shell, where as the latter refers to the transition from M2,3 shell to K Shell. Figure 3 provides a visual description the x-ray emission process [1].

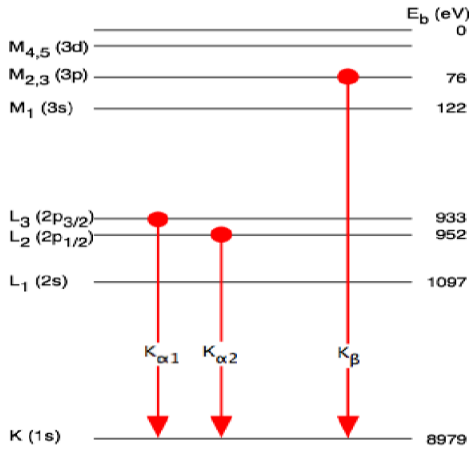

Figure 3: X-Ray emission for copper [1]

Analysis Applied

This section discussed the formulae and methodologies applied to analyze the collected data for corresponding sections.

- X-Ray characteristics of copper

  Using gaussian fit of the data the maximum angle and the uncertainty on it was obtained. Using those values the 'd' value for the corresponding peak was calculated by using equation 2, Bragg's law. In equation 4, the n represents the order of peaks and λ represents the wavelength due to which the peak was caused. Bellow equation 2 has been rearranged as equation 4 along with the formulae used to calculate the uncertainty on 'd.'

$$d = \frac{n\lambda}{2\,sin\theta}\,;\, \sigma_d = \frac{n\lambda\cos\theta\sigma_\theta}{2\sin\theta} \tag{4}$$

  Using the value of 'd' the value of lattice constant 'a' was calculated by using equation 3. The h, k, l variable in equation 3 represents the miller indices of the crystal being used. Below equation 3 has been written again along with the formula used to calculate the uncertainty on the lattice constant.

$$a = d\sqrt{h^2+k^2+l^2};\ \sigma_a = \sigma_d\sqrt{h^2+k^2+l^2}$$

- Laue Method

  Using the distance from the center of each spot 'L' and the distance 'D = 23mm' between the crystal and the photographic film the angle at which the spot is observed is calculated using equation 5. Further, 'd' values are calculated using a rearrangement of equation 3 and those values are then applied to equation 2 to obtain the wavelength that results in each spot, n=1, is used for that.

$$\theta = \frac{1}{2}\arctan\frac{L}{D}\,;\, \sigma_\theta = \frac{\sqrt{(D\sigma_L)^2+(L\sigma_D)^2}}{2\,(D^2+L^2)} \tag{5}$$

- Powder Method

  For the powder method 'd' values are calculated using equation 4 and rearranging it. Thus calculated 'd' values are plotted against a range of lattice constants to get a plot for possible spectrum and the observed spectrum is matched to obtain the lattice constant of the given sample.

APPARATUS

This section lists the apparatus used for collecting data for x-ray diffraction experiment experiments.

List of apparatus

- Sodium Chloride (NaCl) crystal Sample in the orientation 100, 110 and 111
- Lithium Fluoride (LiF) Crystal Sample
- PHYWE X-Ray Machine with copper (Cu) X-ray source
- Photographic films

- Goniometer
- Camera for Powder method
- X-ray machine for Powder method

PROCEDURE

This section discusses the procedure followed to collect the data for three parts of the x-ray diffraction experiment.

X-Ray Characteristics of Copper

The Goniometer was set up inside the PHYWE X-ray machine. Which was connected to the computer, thus collecting the backscatter data from the goniometer at an angle range from 3 degrees to 60 degrees. The X-ray machine was set up at 1mA and 35kV. The pin hole used had a diameter of 2 mm and the diffracting material used was Sodium Chloride crystals of the orientation 100, 110, 111. Figure 4 shows the setup used.

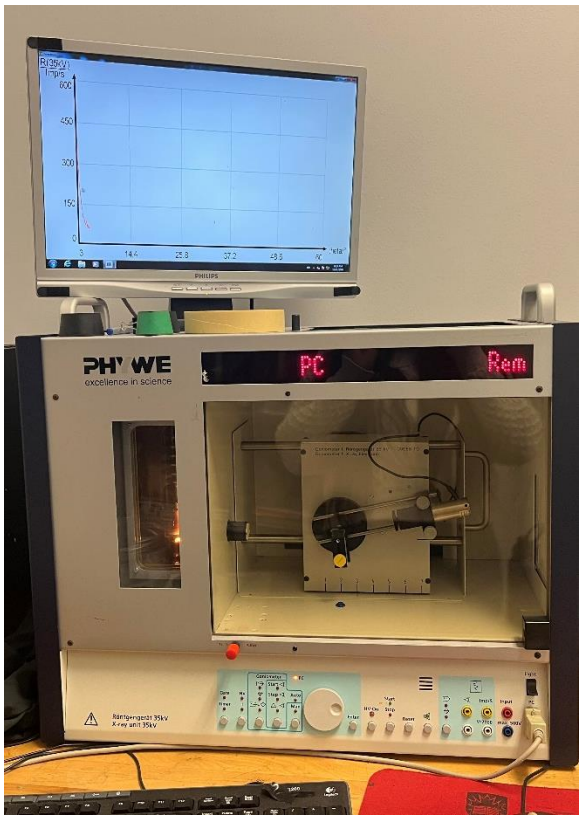

Figure 4: the PHYWE X-Ray Machine with Goniometer

Laue Method

For Laue method the PHYWE X-ray machine was used, the goniometer was removed from it and lithium fluoride (LiF) crystal sample was inserted on top of the pin hole. A Photographic film was setup 23 mm from the sample. A was developed later. Figure 5 depicts the film developed. There

were certain issues encountered during the development process such as the film falling on the ground, which resulted in the film being a bit blotched.

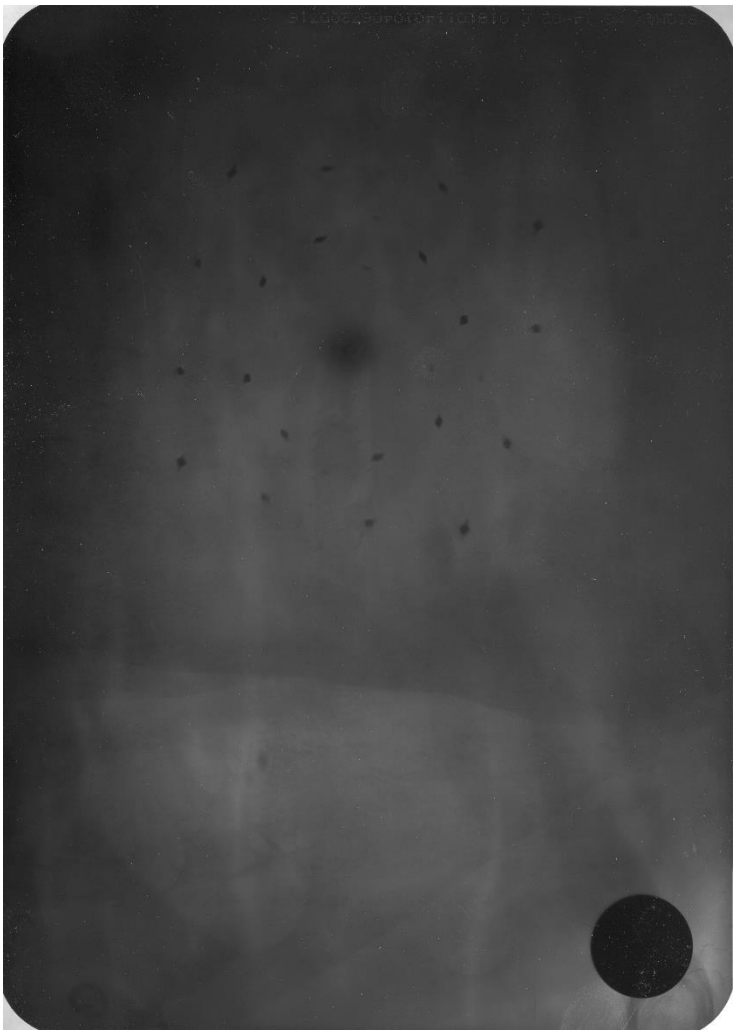

Figure 5: Photographic Film for Laue Method

Powder Method

For the powder method the sample used was 'Sample -H' a body centered crystal (BCC). It was centered using a clay putty. After setting up the sample the photographic film was inserted in the camera, the camera was set up on the x-ray machine for 6 hours. After which the film was removed and developed. The developed film can be seen in figure 6.

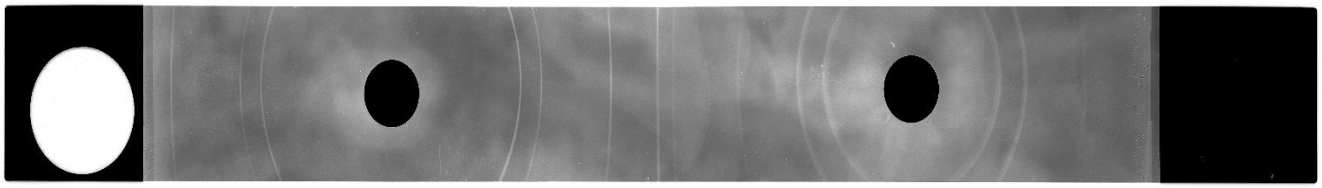

Figure 6: Photographic Film for Powder Method

RESULTS

X-Ray Characteristics of Copper

This section contains the multiple plots obtained from the x-ray counts for the back scatter obtained from the NaCl diffraction for crystal orientations 100, 110, 111. Figure 7 contains the plot from the backscatter of crystal 100. Figures 8 and 9 contain the gauss fit of the n=1 and n=2 from figure 7. Figure 10 contains the plot from the backscatter of crystal 110. Figures 11 and 12 contain the gauss fit of the n=1 and n=2 from figure 10. Figure 13 contains the plot from the backscatter of crystal 111. Figures 14 and 15 contain the gauss fit of the n=1 for the alpha peak and beta peak, figure 16 contains the gaussian fit for n = 2.

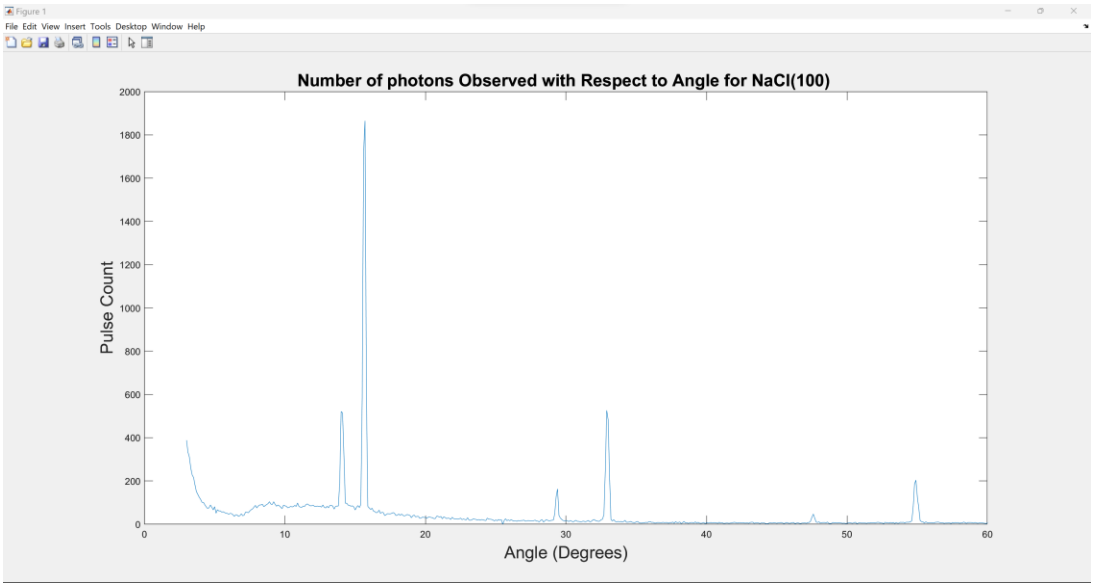

Figure 7: Backscatter count for NaCl Crystal 100

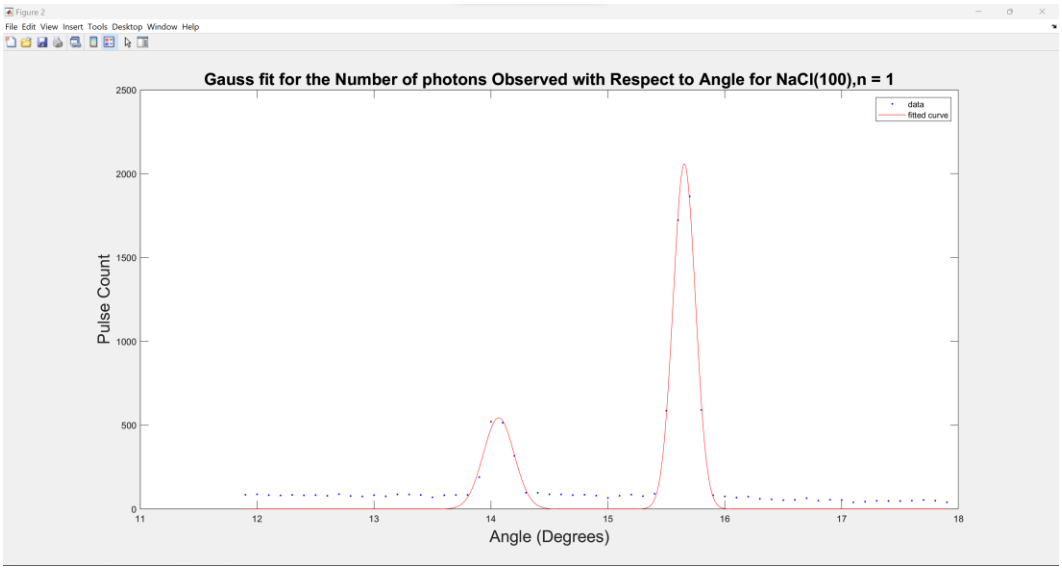

Figure 8: Gaussian fit for data at n = 1, from figure n

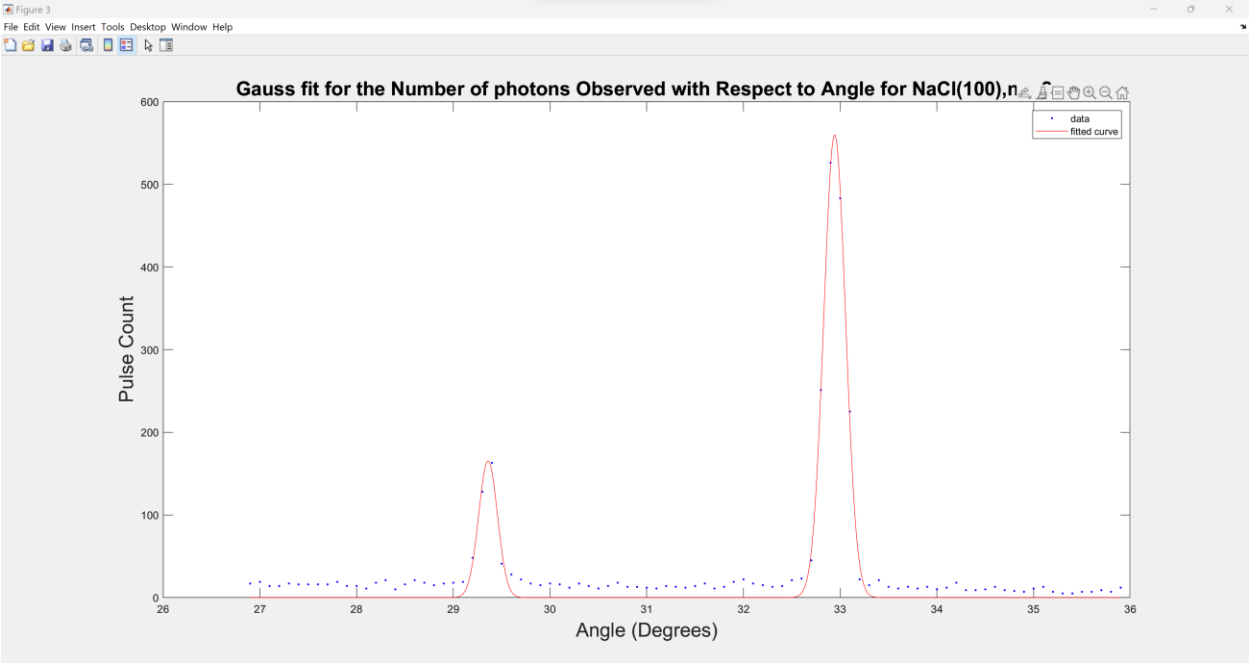

Figure 9: Gaussian fit for data at n = 2, from figure n

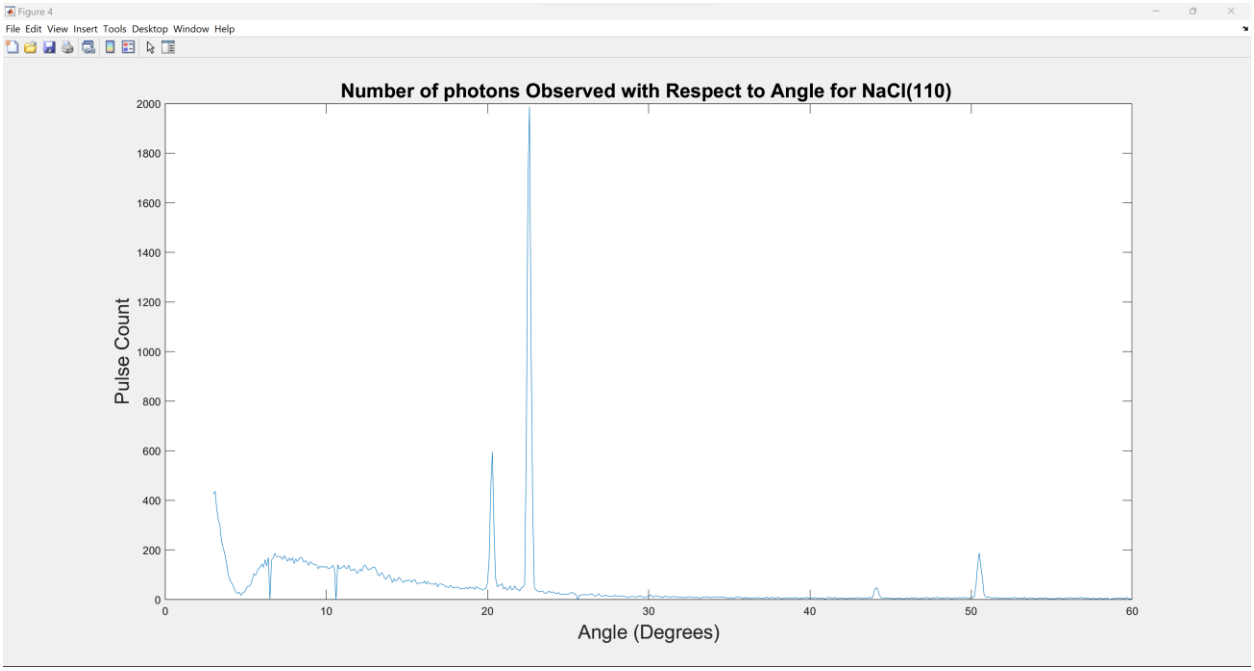

Figure 10: Backscatter count for NaCl Crystal 110

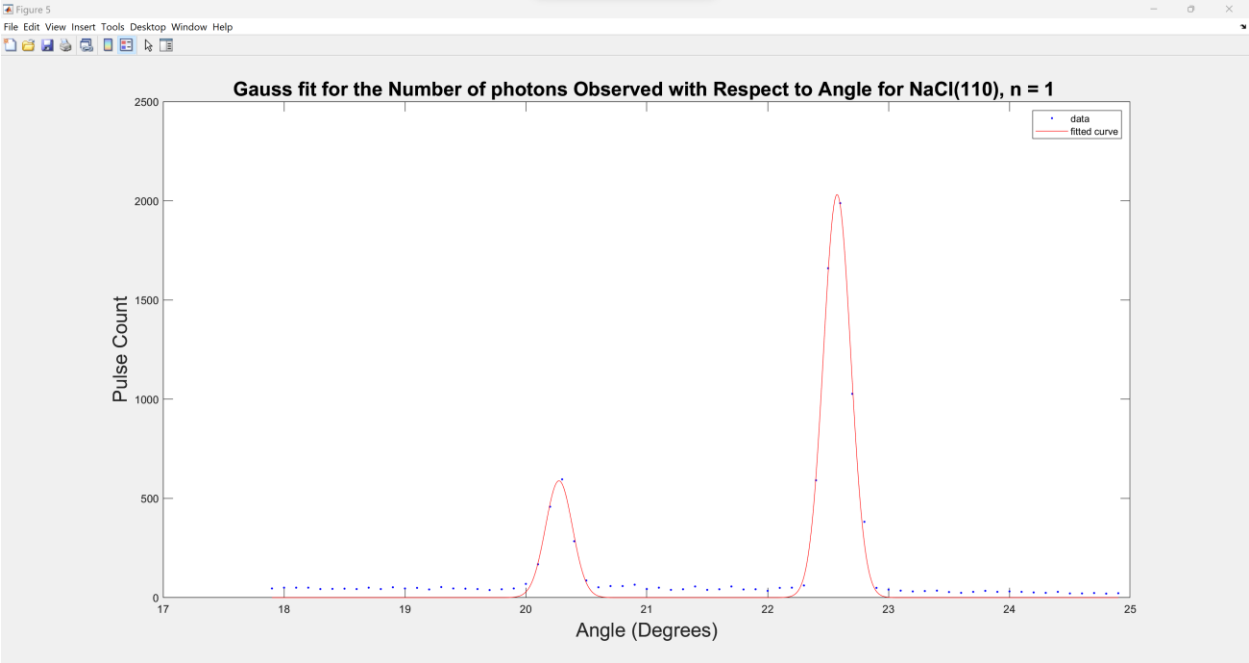

Figure 11: Gaussian fit for data at n = 1, from figure n

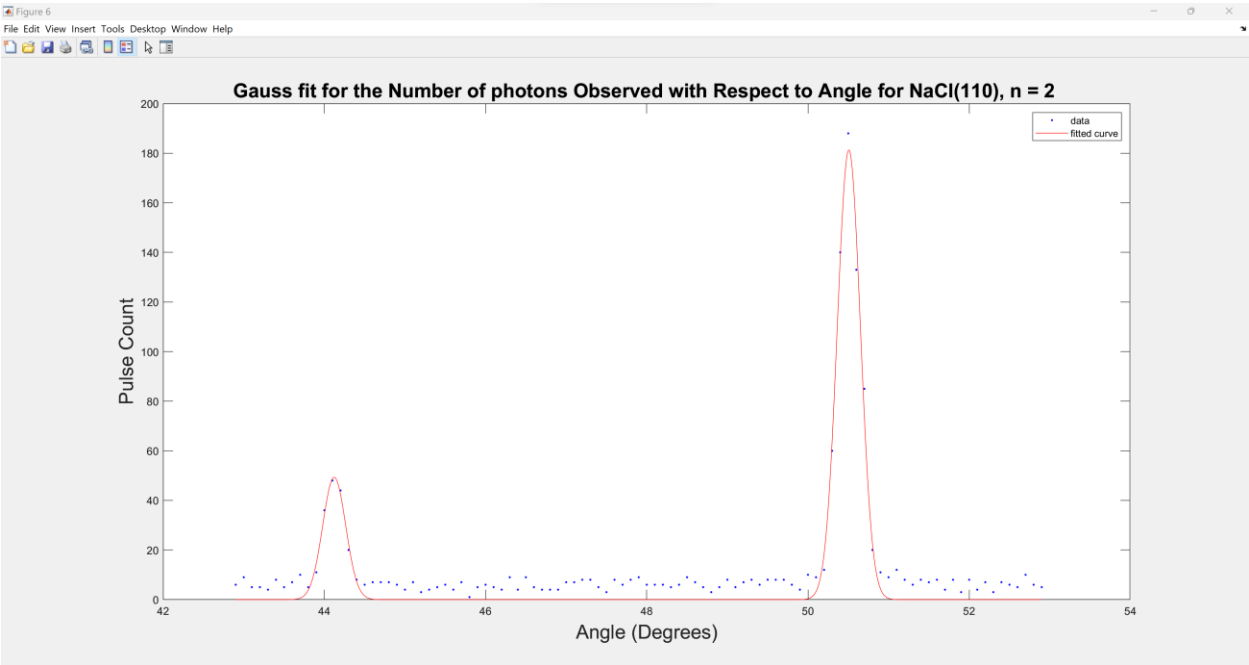

Figure 12: Gaussian fit for data at n = 2, from figure n

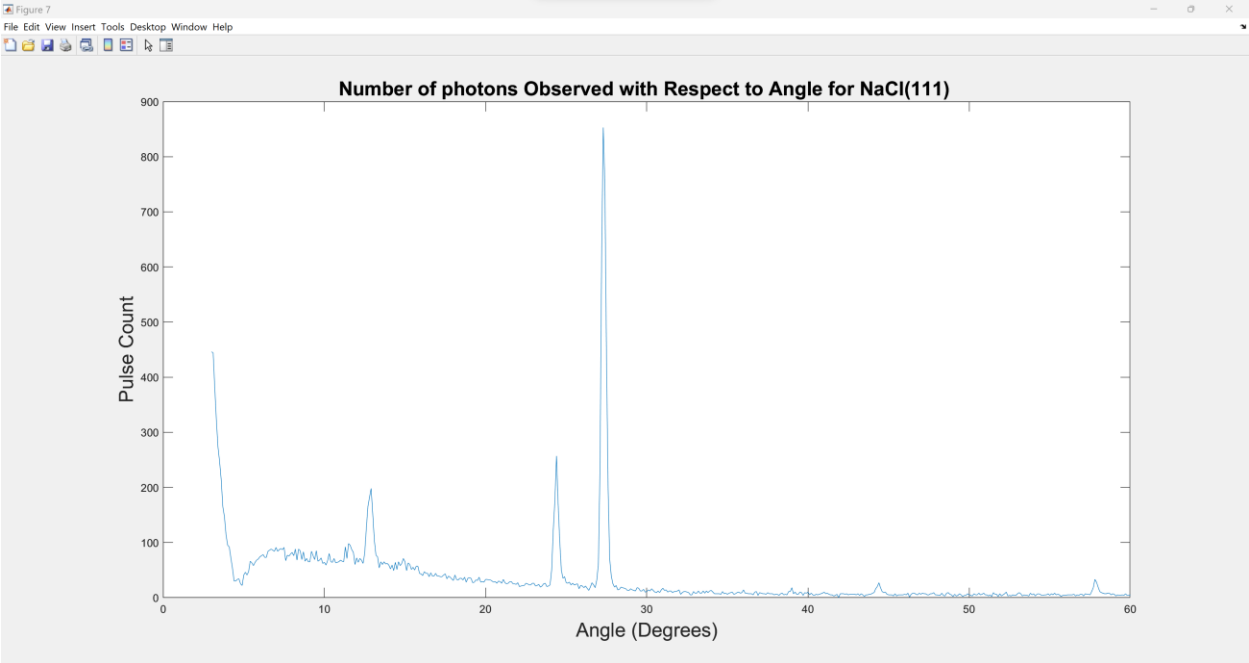

Figure 13: Backscatter count for NaCl Crystal 111

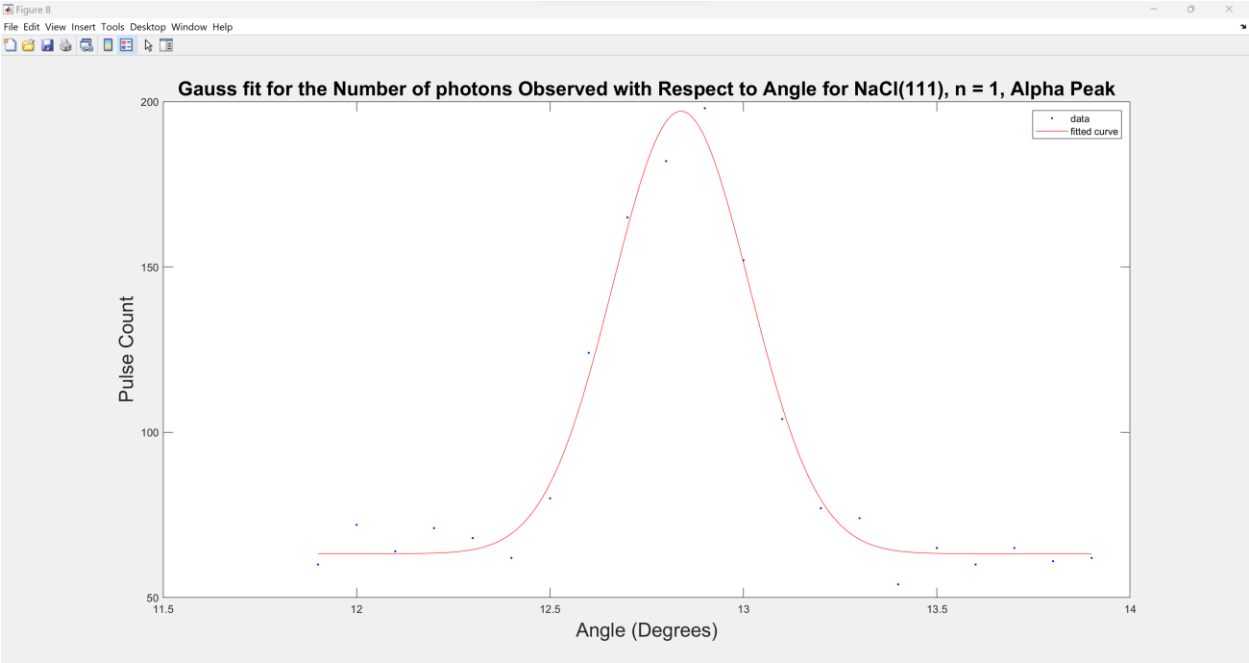

Figure 14: Gaussian fit for data at n = 1 (Alpha peak), from figure n

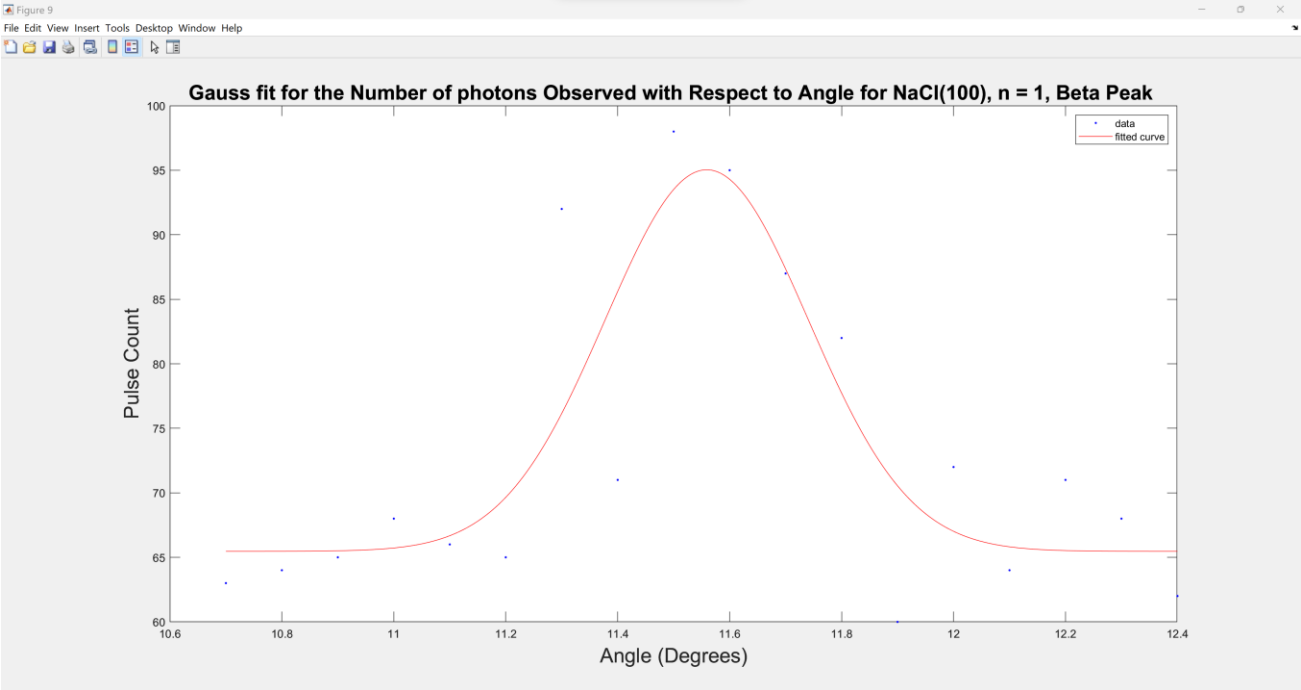

Figure 15: Gaussian fit for data at n = 1 (Beta peak), from figure n

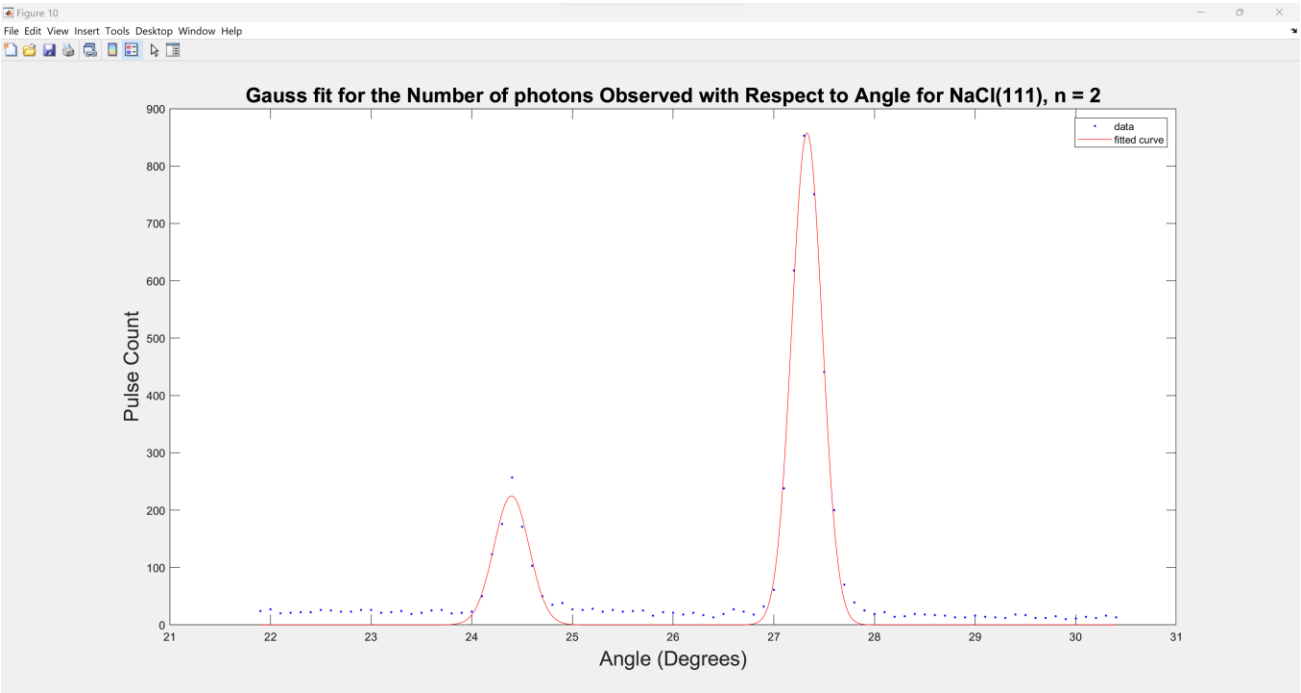

Figure 16: Gaussian fit for data at n = 2, from figure n

From the above plots the value of angles at peak and uncertainty on it was obtained and analyzed resulting in data calculated as shown in table 1. From ten data in table 1 the average value of the lattice constant (a) is calculated to be 5.7601+/- 0.0051 ($\dot{A}$).

Table 1: Data Analyzed for X-Ray Characteristics to calculate the Lattice constant of NaCl

	hkl	Θ α (degree)	d-Θ-α (Å)	a- α (Å)	Θ β (degree)	d-Θ-β (Å)	a- β (Å)	d avg (Å)	a avg (Å)
100									
n=1	002	15.65+/-0.13	2.8588	5.7175	14.07+/-0.19	2.8648	5.7297	2.862+/-0.002	5.723+/-0.044
n=2	002	32.94+/-0.16	2.8369	5.6738	29.36+/-0.14	2.8403	5.6807	2.8386+/-0.0086	5.677+/-0.017
110									
n=1	022	22.57+/-0.16	2.0072	5.6765	20.27+/-0.16	2.0069	5.6765	2.007+/-0.010	5.677+/-0.028
n=2	022	50.51+/-0.20	1.9991	5.6543	44.12+/-0.20	2.0003	5.6576	1.9997+/-0.0047	5.656+/-0.013
111									
n=1	111	12.84+/-0.25	3.4717	6.0186	11.56+/-0.26	3.4748	6.0186	3.473+/-0.050	6.018+/-0.087
n=2	111	27.33+/-0.21	3.3602	5.8201	24.39+/-0.25	3.3717	5.84	3.366+/-0.020	5.830+/-0.035

Laue Method

This method was used to calculate the wavelength that causes diffractions in LiF crystals. The distance of diffraction spots from the center were measured and used. Table 2 depicts the measurement analysis.

Table 2: Data Analyzed for Laue Method for LiF

Spot No.	y (mm)	x (mm)	L (mm)	Θ(exp) (degree)	hkl	Θ(cal) (degree)	k/l	y/x	d (Å)	λ(exp) (Å)	λ (cal) (Å)	T-Test
1	6.26+/-0.35	18.64+/-0.20	19.67+/-0.17	18.5+/-1.1	113	17.548	0.3	0.336+/-0.019	1.59+/-0.35	1.01+/-0.23	0.96+/-0.21	0.17
2	17.54+/-0.61	33.39+/-0.37	37.72+/-0.22	27.7+/-1.0	204	26.565	0	0.525+/-0.019	1.18+/-0.26	1.10+/-0.25	1.06+/-0.11	0.15
3	13.38+/-0.55	28.44+/-0.31	31.43+/-0.12	25.2+/-1.1	224	24.095	0.5	0.471+/-0.020	1.08+/-0.24	0.92+/-0.21	0.75+/-0.39	0.38
4	3.45+/-0.22	13.62+/-0.16	14.05+/-0.15	14.20+/-0.93	133	13.263	1	0.253+/-0.017	1.21+/-0.27	0.59+/-0.14	0.56+/-0.47	0.08
5	8.02+/-0.41	21.32+/-0.22	22.78+/-0.16	20.6+/-1.1	244	19.471	1	0.376+/-0.020	0.88+/-0.20	0.62+/-0.14	0.59+/-0.34	0.09

The last three columns of table 2 represent the experimentally calculated values of the wavelength and the numerically calculated values of wavelength along with the T-test values for the corresponding spots. For the given spots the T-test values for all the five spots are less than two hence the values are acceptable. The calculation was only done for 5 spots because of the limited visibility of the photographic film.

Powder Method

This method was used to analyze iron (Fe) BCC crystal sample, 'sample H'. The sample was exposed to X-ray radiation from a copper source inside a circular camera whose rim was covered in a circular photographic film. The Spectral lines obtained from the film in figure 6 were used to calculate the crystal orientations that result spectral lines. The lattice constant for iron is 2.866 Å. Figure 17 depicts the spectral lines obtained along with the multiple spectrums that could have had been possible. This result is not the most appealing and depicts high uncertainty. Most reasons for such uncertainty could be huge variability of measurements taken using ImageJ software, high sensitivity of the apparatus used, and issues encountered during the photographic development process [3].

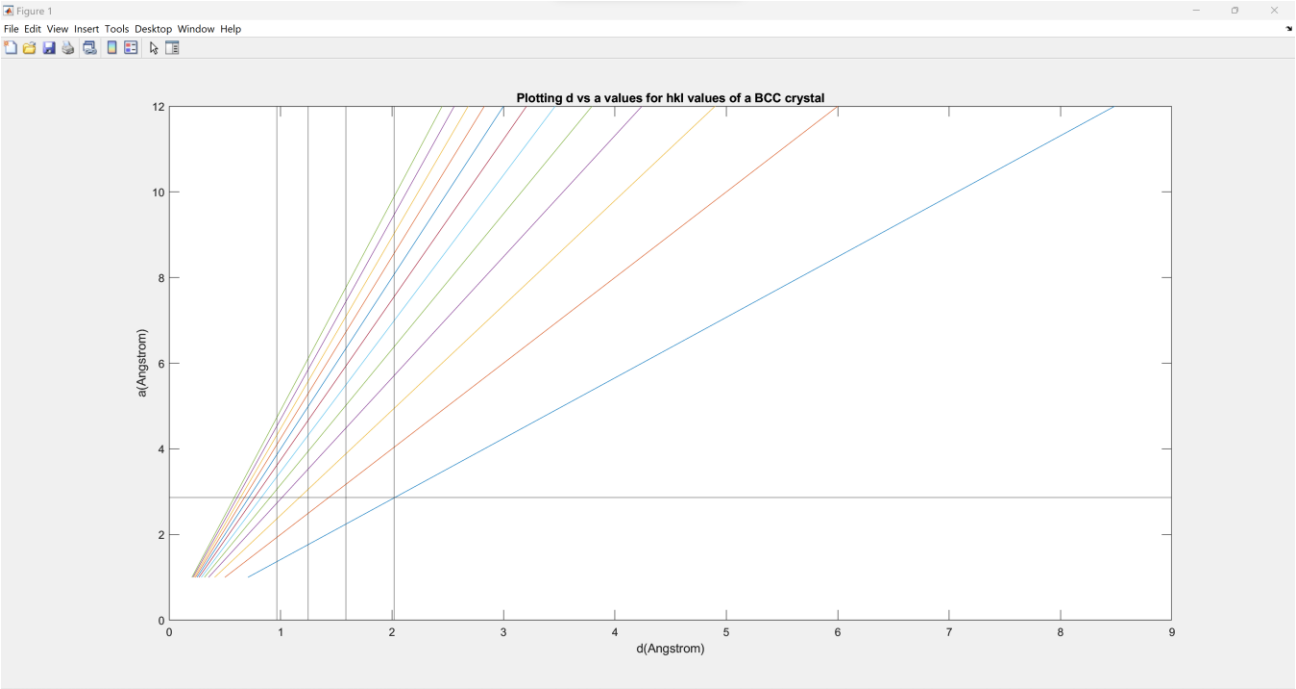

Figure 17: Spectral Range for BCC Crystals and Spectrum Obtained

DISCUSSION

This experiment used PHYWE X-Ray Machine with copper (Cu) X-ray source to analyze various crystal structures. For the first part of the experiment a goniometer was set up inside the PHYWE X-Ray Machine. The goniometer rotated crystal sample at an angle range between 3° and 60°. The type of NaCl Crystal samples were used for this part and analyzed separately, their sample had miller indices (100, 110, 111). The lattice constant was analyzed for each of the three and sample and was measured to be (5.700 +/- 0.047 $\dot{A}$), (5.667 +/- 0.031 $\dot{A}$) and (5.924 +/- 0.094 $\dot{A}$) respectively. The final average value of the lattice constant calculated was (5.7601+/- 0.0051 $\dot{A}$). Which is with the acceptable range of values for such. For the second part of the experiment the goniometer was removed from inside the X-ray machine and a photographic film stand was placed inside it. The film was set up at 23mm from the x ray source which was covered by a LiF crystal sample. The photographic film was exposed to the setup of a time period of 1 hour after which it was developed and analyzed, then the spots on the film which represent major diffraction patterns were used to calculate the wavelengths of the x-ray spectrum that resulted in those spots and were further compared with the expected values of such wavelength. A T-test was performed on the values which gave the values less than 2 for the wavelength thus deeming the results acceptable. The wavelengths were only calculated for 5 spots as the more spots were not very visible on the film. For the final part of the experiment an unknown BCC sample was choose, 'Sample H.' It was centered and setup inside a circular camera with clay putty. And a circular photographic film was setup indie the camera. The setup was exposed to copper X-rays for 6 hours. Spectral lines were observed on the photographic film. The spectrum was analyzed to calculate the lattice constant of the unknown substance which was calculated to be around 2.866 $\dot{A}$. This is the lattice constant for Iron which was then identified as the sample.

The uncertainties in the values for part 1. The obtained plot for the number of backscatter x-rays obtained was plotted against the angle of observation at which they were observed. The data obtained was analyzed by using gaussian curves and thus measuring the angle at which maximum number of x-ray backscatter was observed and the uncertainty on it. The uncertainty due to the gaussian curve was the only error source for this calculation. For Laue and powder method ImageJ software was used which does not provide high resolution for measurements and hence was a huge source of error. There were also some issues encountered while developing the photographic films such as one of the films dropped on the floor and due to no prior experience of having developed a film there was some time between transitioning from solution to solution which should have not been done hence resulted in darker and unclear films being developed.

Some of the major application of X-Ray diffraction is identification of unknown crystalline materials. Some of the techniques used in this experiment are directly applicable in the field of minerology and geology. X-Ray diffraction is also used for quality control for polymer materials. It helps analyze the material and determine if it fits the health and safety standard of the final product being built. X-rays are also a huge part of the medical industry to help analyze the bone structure of an individual [4].

Bibliography

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APPPENDIX I: MATLAB Code: X-Ray Characteristics Copper

```
% NaCl_100, hkl = 002
Theta100 = NaCl100(:,1);
Theta100 = table2array(Theta100);

Imp_s_100 = NaCl100(:,2);
Imp_s_100 = table2array(Imp_s_100);

lamda_b = (12398/8903)*(10^(-10));
lamda_a = (12398/8036.5)*(10^(-10));

figure(1)
plot(Theta100, Imp_s_100)
xlabel('Angle (Degrees)','FontSize',18);
ylabel('Pulse Count','FontSize',18);
title ('Number of photons Observed with Respect to Angle for NaCl(100)
','FontSize',18)

% For then gauss fit
    %n = 1
    figure(2)
    Theta_100_1 = Theta100(90:150,1);
    Imp_s_100_1 = Imp_s_100(90:150,1);
    [F_100_1, gof_100_1, fit_output_100_1] = fit( Theta_100_1, Imp_s_100_1,
'gauss2');
    plot( F_100_1,Theta_100_1, Imp_s_100_1);
    xlabel('Angle (Degrees)','FontSize',18);
    ylabel('Pulse Count','FontSize',18);
    title ('Gauss fit for the Number of photons Observed with Respect to Angle for
NaCl(100),n = 1','FontSize',18)


    %n = 2
    figure(3)
    Theta_100_2 = Theta100(240:330,1);
    Imp_s_100_2 = Imp_s_100(240:330,1);
    [F_100_2,gof_100_2,fit_output_100_2] = fit( Theta_100_2, Imp_s_100_2, 'gauss2');
    plot( F_100_2,Theta_100_2, Imp_s_100_2);
    xlabel('Angle (Degrees)','FontSize',18);
    ylabel('Pulse Count','FontSize',18);
    title ('Gauss fit for the Number of photons Observed with Respect to Angle for
NaCl(100),n = 2 ','FontSize',18)

% Calculation spacing between two faces 'd'
% First set of peaks, n=1
% Beta
thetamax100b1 = (F_100_1.b2*pi)/180;  %In radians
d_b100_1 = lamda_b/(2*sin(thetamax100b1)); % d = ((n*lamda)/(2*sin(theta)))
b_100_1 = d_b100_1*2; % a = d*sqrt((h*h)+(k*k)+(l*l))
    %Error Analysis
    err_Theta_rad_100b1 = (F_100_1.c2*pi)/180;
```

```
    err_d_b100_1 =
err_Theta_rad_100b1*((lamda_b*cos(thetamax100b1))/(2*sin(thetamax100b1)*sin(thetamax1
00b1)));
    err_b_100_1 = 2*err_d_b100_1;

%Alpha
thetamax100a1 = (F_100_1.b1*pi)/180;  %In radians
d_a100_1 = (lamda_a)/(2*sin(thetamax100a1));
a_100_1 = d_a100_1*2; %a = d*sqrt((h*h)+(k*k)+(l*l))
    %Error Analysis
    err_Theta_rad_100a1 = (F_100_1.c1*pi)/180;
    err_d_a100_1 =
err_Theta_rad_100a1*((lamda_a*cos(thetamax100a1))/(2*sin(thetamax100a1)*sin(thetamax1
00a1)));
    err_a_100_1 = 2* err_d_a100_1;

% Mean error
    err_d_100_1 = (sqrt((err_d_a100_1*err_d_a100_1)+(err_d_b100_1*err_d_b100_1)))/2;
    err_ab_100_1 = (sqrt((err_a_100_1*err_a_100_1)+(err_b_100_1*err_b_100_1)))/2;

% Second set of peaks, n=2
% Beta
thetamax100b_2 = (F_100_2.b2*pi)/180;  %In radians
d_b100_2 = (2*lamda_b)/(2*sin(thetamax100b_2)); % d = ((n*lamda)/(2*sin(theta)))
b_100_2 = d_b100_2*2; %a = d*sqrt((h*h)+(k*k)+(l*l))
    %Error Analysis
    err_Theta_rad_100b2 = (F_100_2.c2*pi)/180;
    err_d_b100_2 =
err_Theta_rad_100b2*((2*lamda_b*cos(thetamax100b_2))/(2*sin(thetamax100b_2)*sin(theta
max100b_2)));
    err_b_100_2 = 2* err_d_b100_2;

%Alpha
% Second set of peaks
thetamax100a_2 = (F_100_2.b1*pi)/180;  %In radians
d_a100_2 = (2*lamda_a)/(2*sin(thetamax100a_2));
a_100_2 = d_a100_2*2; %a = d*sqrt((h*h)+(k*k)+(l*l))
    %Error Analysis
    err_Theta_rad_100a2 = (F_100_2.c1*pi)/180;
    err_d_a100_2 =
err_Theta_rad_100a2*((2*lamda_a*cos(thetamax100a_2))/(2*sin(thetamax100a_2)*sin(theta
max100a_2)));
    err_a_100_2 = 2* err_d_a100_2;

% Mean error
err_d_100_2 = (sqrt((err_d_a100_2*err_d_a100_2)+(err_d_b100_2*err_d_b100_2)))/2;
err_ab_100_2 = (sqrt((err_a_100_2*err_a_100_2)+(err_b_100_2*err_b_100_2)))/2;
%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
% NaCl_110, hkl = 022
Theta110 = NaCl110(:,1);
Theta110 = table2array(Theta110);

Imp_s_110 = NaCl110(:,2);
Imp_s_110 = table2array(Imp_s_110);
```

```
lamda_b = (12398/8903)*(10^(-10));
lamda_a = (12398/8036.5)*(10^(-10));

figure(4)
plot(Theta110, Imp_s_110)
xlabel('Angle (Degrees)','FontSize',18);
ylabel('Pulse Count','FontSize',18);
title ('Number of photons Observed with Respect to Angle for NaCl(110)
','FontSize',18)


% For then gauss fit
    %n = 1
    figure(5)
    Theta_110_1 = Theta110(150:220,1);
    Imp_s_110_1 = Imp_s_110(150:220,1);
    [F_110_1, gof_110_1, fit_output_110_1] = fit( Theta_110_1, Imp_s_110_1,
'gauss2');
    plot( F_110_1,Theta_110_1, Imp_s_110_1);
    xlabel('Angle (Degrees)','FontSize',18);
    ylabel('Pulse Count','FontSize',18);
    title ('Gauss fit for the Number of photons Observed with Respect to Angle for
NaCl(110), n = 1 ','FontSize',18)

    %n = 2
    figure(6)
    Theta_110_2 = Theta110(400:500,1);
    Imp_s_110_2 = Imp_s_110(400:500,1);
    [F_110_2,gof_110_2,fit_output_110_2] = fit( Theta_110_2, Imp_s_110_2, 'gauss2');
    plot( F_110_2,Theta_110_2, Imp_s_110_2);
    xlabel('Angle (Degrees)','FontSize',18);
    ylabel('Pulse Count','FontSize',18);
    title ('Gauss fit for the Number of photons Observed with Respect to Angle for
NaCl(110), n = 2 ','FontSize',18)


% Calculation spacing between two faces 'd'
% First set of peaks, n=1
% Beta
root8 = sqrt(8);
thetamax110b1 = (20.3*pi)/180;  %In radians
d_b110_1 = lamda_b/(2*sin((thetamax110b1))); % d = ((n*lamda)/(2*sin(theta)))
b_110_1 = d_b110_1*root8; % a = d*sqrt((h*h)+(k*k)+(l*l))
    %Error Analysis
    err_Theta_rad_110b1 = (F_110_1.c2*pi)/180;
    err_d_b110_1 =
err_Theta_rad_110b1*((lamda_b*cos(thetamax110b1))/(2*sin(thetamax110b1)*sin(thetamax1
10b1)));
    err_b_110_1 = root8*err_d_b110_1;

%Alpha
thetamax110a1 = (22.6*pi)/180;  %In radians
d_a110_1 = (lamda_a)/(2*sin((thetamax110a1)));
```

```
a_110_1 = d_b110_1*root8; %a = d*sqrt((h*h)+(k*k)+(l*l))
    %Error Analysis
    err_Theta_rad_110a1 = (F_110_1.c1*pi)/180;
    err_d_a110_1 =
err_Theta_rad_110a1*((lamda_a*cos(thetamax110a1))/(2*sin(thetamax110a1)*sin(thetamax1
10a1)));
    err_a_110_1 = root8*err_d_a110_1;

% Mean error
err_d_110_1 = (sqrt((err_d_a110_1*err_d_a110_1)+(err_d_b110_1*err_d_b110_1)))/2;
err_ab_110_1 = (sqrt((err_a_110_1*err_a_110_1)+(err_b_110_1*err_b_110_1)))/2;

% Second set of peaks, n=2
% Beta
root2 = sqrt(2);
thetamax110b_2 = (F_110_2.b2*pi)/180;  %In radians
d_b110_2 = (2*lamda_b)/(2*sin(thetamax110b_2)); % d = ((n*lamda)/(2*sin(theta)))
b_110_2 = d_b110_2*root8; % a = d*sqrt((h*h)+(k*k)+(l*l))
    %Error Analysis
    err_Theta_rad_110b2 = (F_110_2.c2*pi)/180;
    err_d_b110_2 =
err_Theta_rad_110b2*((2*lamda_b*cos(thetamax110b_2))/(2*sin(thetamax110b_2)*sin(theta
max110b_2)));
    err_b_110_2 = root8*err_d_b110_2;

%Alpha
% Second set of peaks
thetamax110a_2 = (F_110_2.b1*pi)/180;  %In radians
d_a110_2 = (2*lamda_a)/(2*sin(thetamax110a_2));
a_110_2 = d_a110_2*root8; %a = d*sqrt((h*h)+(k*k)+(l*l))
    %Error Analysis
    err_Theta_rad_110a2 = (F_110_2.c1*pi)/180;
    err_d_a110_2 =
err_Theta_rad_110a2*((2*lamda_a*cos(thetamax110a_2))/(2*sin(thetamax110a_2)*sin(theta
max110a_2)));
    err_a_110_2 = root8*err_d_a110_2;

% Mean error
err_d_110_2 = (sqrt((err_d_a110_2*err_d_a110_2)+(err_d_b110_2*err_d_b110_2)))/2;
err_ab_110_2 = (sqrt((err_a_110_2*err_a_110_2)+(err_b_110_2*err_b_110_2)))/2;
% %%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
%
% NaCl_111
Theta111 = NaCl111(:,1);
Theta111 = table2array(Theta111);

Imp_s_111 = NaCl111(:,2);
Imp_s_111 = table2array(Imp_s_111);

figure(7)
plot(Theta111, Imp_s_111)
ft = fittype("a1*exp(-((x-b1)/c1)^2)+d1");
xlabel('Angle (Degrees)','FontSize',18);
ylabel('Pulse Count','FontSize',18);
```

```
title ('Number of photons Observed with Respect to Angle for NaCl(111)
','FontSize',18)

% For then gauss fit
    %n = 1
    % alpha
    figure(8)
    Theta_a111_1 = Theta111(90:110,1);
    Imp_s_a111_1 = Imp_s_111(90:110,1);
    [F_a111_1, gof_a111_1, fit_output_a111_1] = fit( Theta_a111_1, Imp_s_a111_1,
ft,"StartPoint",[140,13,1,60]);
    plot( F_a111_1,Theta_a111_1, Imp_s_a111_1);
    xlabel('Angle (Degrees)','FontSize',18);
    ylabel('Pulse Count','FontSize',18);
    title ('Gauss fit for the Number of photons Observed with Respect to Angle for
NaCl(111), n = 1, Alpha Peak','FontSize',18)

    % beta
    figure(9)
    Theta_b111_1 = Theta111(78:95,1);
    Imp_s_b111_1 = Imp_s_111(78:95,1);
    [F_b111_1, gof_b111_1, fit_output_b111_1] = fit( Theta_b111_1, Imp_s_b111_1,
ft,"StartPoint",[100,11.5,0.2,60]);
    plot( F_b111_1,Theta_b111_1, Imp_s_b111_1);
    xlabel('Angle (Degrees)','FontSize',18);
    ylabel('Pulse Count','FontSize',18);
    title ('Gauss fit for the Number of photons Observed with Respect to Angle for
NaCl(100), n = 1, Beta Peak ','FontSize',18)

    %n = 2
    figure(10)
    Theta_111_2 = Theta111(190:275,1);
    Imp_s_111_2 = Imp_s_111(190:275,1);
    [F_111_2,gof_111_2,fit_output_111_2] = fit( Theta_111_2, Imp_s_111_2, 'gauss2');
    plot( F_111_2,Theta_111_2, Imp_s_111_2);
    xlabel('Angle (Degrees)','FontSize',18);
    ylabel('Pulse Count','FontSize',18);
    title ('Gauss fit for the Number of photons Observed with Respect to Angle for
NaCl(111), n = 2','FontSize',18)

% Calculation spacing between two faces 'd'
% First set of peaks, n=1
% Beta
root3 = sqrt(3);
thetamax111b1 = (F_b111_1.b1*pi)/180;  %In radians
d_b111_1 = lamda_b/(2*sin(thetamax111b1)); % d = ((n*lamda)/(2*sin(theta)))
b_111_1 = d_b111_1*root3; % a = d*sqrt((h*h)+(k*k)+(l*l))
        %Error Analysis
        err_Theta_rad_111b1 = (F_b111_1.c1*pi)/180;
        err_d_b111_1 =
err_Theta_rad_111b1*((lamda_b*cos(thetamax111b1))/(2*sin(thetamax111b1)*sin(thetamax1
11b1)));
        err_b_111_1 = root3*err_d_b111_1;
```

```
%Alpha
thetamax111a1 = (F_a111_1.b1*pi)/180;  %In radians
d_a111_1 = (lamda_a)/(2*sin(thetamax111a1));
a_111_1 = d_b111_1*root3; %a = d*sqrt((h*h)+(k*k)+(l*l))
        %Error Analysis
        err_Theta_rad_111a1 = (F_a111_1.c1*pi)/180;
        err_d_a111_1 =
err_Theta_rad_111a1*((lamda_a*cos(thetamax111a1))/(2*sin(thetamax111a1)*sin(thetamax1
11a1)));
        err_a_111_1 = root3*err_d_a111_1;

% Mean error
err_d_111_1 = (sqrt((err_d_a111_1*err_d_a111_1)+(err_d_b111_1*err_d_b111_1)))/2;
err_ab_111_1 = (sqrt((err_a_111_1*err_a_111_1)+(err_b_111_1*err_b_111_1)))/2;

% Second set of peaks, n=2
% Beta
root3 = sqrt(3);
thetamax111b_2 = (F_111_2.b2*pi)/180;  %In radians
d_b111_2 = (2*lamda_b)/(2*sin(thetamax111b_2)); % d = ((n*lamda)/(2*sin(theta)))
b_111_2 = d_b111_2*root3; % a = d*sqrt((h*h)+(k*k)+(l*l))
        %Error Analysis
        err_Theta_rad_111b2 = (F_111_2.c2*pi)/180;
        err_d_b111_2 =
err_Theta_rad_111b2*((2*lamda_b*cos(thetamax111b_2))/(2*sin(thetamax111b_2)*sin(theta
max111b_2)));
        err_b_111_2 = root3*err_d_b111_2;

%Alpha
% Second set of peaks
thetamax111a_2 = (F_111_2.b1*pi)/180;  %In radians
d_a111_2 = (2*lamda_a)/(2*sin(thetamax111a_2));
a_111_2 = d_a111_2*root3; %a = d*sqrt((h*h)+(k*k)+(l*l))
        %Error Analysis
        err_Theta_rad_111a2 = (F_111_2.c1*pi)/180;
        err_d_a111_2 =
err_Theta_rad_111a2*((2*lamda_a*cos(thetamax111a_2))/(2*sin(thetamax111a_2)*sin(theta
max111a_2)));
        err_a_111_2 = root3*err_d_a111_2;

% Mean error
err_d_111_2 = (sqrt((err_d_a111_2*err_d_a111_2)+(err_d_b111_2*err_d_b111_2)))/2;
err_ab_111_2 = (sqrt((err_a_111_2*err_a_111_2)+(err_b_111_2*err_b_111_2)))/2;
```

APPPENDIX I: MATLAB Code: Powder Method

```
a = linspace(1,12,12);
%d = linspace(0,20,21);
hkl = [1 1 0; 2 0 0; 2 1 1; 2 2 0; 3 1 0;2 2 2; 3 2 1; 4 0 0; 4 1 1; 3 3 0;
    4 2 0; 3 3 2];
hklsqrd = hkl.^2;
d = [0.707106781    0.5    0.40824829    0.353553391  0.316227766  0.288675135
      0.267261242  0.25   0.23570226    0.223606798  0.213200716  0.204124145
1.414213562  1      0.816496581  0.707106781  0.632455532  0.577350269  0.534522484
      0.5    0.471404521  0.447213595  0.426401433  0.40824829
2.121320344  1.5    1.224744871  1.060660172  0.948683298  0.866025404  0.801783726
      0.75   0.707106781  0.670820393  0.639602149  0.612372436
2.828427125  2      1.632993162  1.414213562  1.264911064  1.154700538  1.069044968
      1      0.942809042  0.894427191  0.852802865  0.816496581
3.535533906  2.5    2.041241452  1.767766953  1.58113883   1.443375673  1.33630621
      1.25   1.178511302  1.118033989  1.066003582  1.020620726
4.242640687  3      2.449489743  2.121320344  1.897366596  1.732050808  1.603567451
      1.5    1.414213562  1.341640786  1.279204298  1.224744871
4.949747468  3.5    2.857738033  2.474873734  2.213594362  2.020725942  1.870828693
      1.75   1.649915823  1.565247584  1.492405014  1.428869017
5.656854249  4      3.265986324  2.828427125  2.529822128  2.309401077  2.138089935
      2      1.885618083  1.788854382  1.705605731  1.632993162
6.363961031  4.5    3.674234614  3.181980515  2.846049894  2.598076211  2.405351177
      2.25   2.121320344  2.01246118   1.918806447  1.837117307
7.071067812  5      4.082482905  3.535533906  3.16227766   2.886751346  2.672612419
      2.5    2.357022604  2.236067977  2.132007164  2.041241452
7.778174593  5.5    4.490731195  3.889087297  3.478505426  3.175426481  2.939873661
      2.75   2.592724864  2.459674775  2.34520788   2.245365598
8.485281374  6      4.898979486  4.242640687  3.794733192  3.464101615  3.207134903
      3      2.828427125  2.683281573  2.558408596  2.449489743
];
 for i = 1:12
     alpha(i) = power((hklsqrd(i,1) + hklsqrd(i,2) + hklsqrd(i,3)), 0.5);
 end

 for i = 1:12
    plot (d(1:12, i),a);

    hold on
 end
 xlabel('d(Angstrom)');
 ylabel('a(Angstrom)');
 title('Plotting d vs a values for hkl values of a BCC crystal');
 yline(2.866);

 xline(2.019228576);
 xline(1.585590743);
 xline(1.248680217);
 xline(0.965719475);
```