

FYSC23

Powder X-ray Diffraction Lab

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February 28, 2025

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1 Introduction

2 Theory

[1]

2.1 Crystal Lattice Structure

To calculate the distance between planes the formula

$$\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}. \quad (1)$$

If we use this formula for a simple cubic with $a = b = c = 0.432 \text{ nm}$ for the plane(111) we get the separation to $d = 0.249 \text{ nm}$. If we do the same for the plane (211) we obtain a separation of $d = 0.176 \text{ nm}$ and for the plane (100) we get $d = 0.432 \text{ nm}$.

2.2 Diffraction

To evaluate diffraction patterns Bragg's law is mainly used, with is defined as

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

where n is an integer, λ is the wavelength of the incoming photon, d is the spacing between the planes and θ is the scattering angle. Using this we see that if the incoming x-ray has a wavelength of 0.07 nm we can at minimum spacing which can be observed is 0.35 \AA . In order to observe smaller spacings, a higher photon energy would have to be used.

For example if we have a (111) plane with an incident angle $\theta = 11.2^\circ$ when X-rays of wavelength of 0.154 nm then the side of the side of each unit cell can be calculated as follows: From Equation 2 we obtain a the distance planer separation to $d = 0.397 \text{ nm}$, from Equation 1 we obtain that a separation of $d = 0.397 \text{ nm}$ leads to a lattice constant of $a = 6.36 \text{ \AA}$ for a simple cubic.

Another example for a orthorhombic unit cell with dimensions $a = 5.74 \text{ \AA}$, $b = 7.96 \text{ \AA}$ and $c = 4.95 \text{ \AA}$ one can calculate the incident angles for the (100), (010), and (111) planes if the wavelength is 0.083 nm . For the (100) plane Equation 1 gives a planar separation of $d_{(100)} = 0.574 \text{ nm}$, (010) gives $d_{(010)} = 0.796 \text{ nm}$ and (111) gives $d_{(111)} = 0.339 \text{ nm}$. Using Equation 2 the following angles are found: $\theta_{(100)} = 4.15^\circ$, $\theta_{(010)} = 2.99^\circ$ and $\theta_{(111)} = 7.03^\circ$.

2.3 Structure Factors

Due to the structure of different types of lattices, the diffraction pattern may change. This is due to structure function which are defined as

$$F(hkl) = \sum_m f_m \exp(2\pi i(u_m h + v_m k + w_m l)). \quad (3)$$

where f_m is the atomic form factor and (u_m, v_m, w_m) are the fractional coordinates of atoms in the unit cell. for a bcc we know that we have the fractional coordinates (0,0,0) for each corner and (1/2,1/2,1/2). and for an fcc we have the (0,0,0) for each corner, (0,1/2,1/2), (1/2,0,1/2) and (1/2,1/2,0). In the equation above h,k,l is the value of the miller index. Putting all of this together we know that we will obtain diffraction peaks only if $F(hkl) \neq 0$ [2].

From this we can evaluate which planes we will see from bcc and fcc. From Equation 3 we can see that the points located at (0,0,0) will always yield a non-zero result. However, if we start looking at the bcc crystal we can obtain the following:

$$F(hkl) = f_m \exp[2\pi i (0h + 0k + 0l)] + f_m \exp\left[2\pi i \left(\frac{h}{2} + \frac{k}{2} + \frac{l}{2}\right)\right] = f_m \left[1 + \exp\left(2\pi i \frac{h+k+l}{2}\right)\right] \quad (4)$$

$$\text{if } F(hkl) \neq 0 \implies -1 \neq \exp\left(2\pi i \frac{h+k+l}{2}\right) \implies \frac{h+k+l}{2} \neq n + \frac{1}{2}, \text{ where } n=1,2,3... \quad (5)$$

$$\implies h+k+l \neq 2n+1. \quad (6)$$

Above we used Euler's identity which says $\exp(i\pi(n+1/2)) + 1 = 0$. We have thus shown that for the bcc structure wont show any planes where $h+k+l$ are odd. For the fcc we get the following instead:

$$F(hkl) = f_m + f_m \exp\left[2\pi i \left(\frac{h}{2} + \frac{k}{2}\right)\right] + f_m \exp\left[2\pi i \left(\frac{h}{2} + \frac{l}{2}\right)\right] + f_m \exp\left[2\pi i \left(\frac{k}{2} + \frac{l}{2}\right)\right] \quad (7)$$

$$\text{if } F(hkl) \neq 0 \implies -1 \neq \exp\left[2\pi i \left(\frac{h}{2} + \frac{k}{2}\right)\right] + \exp\left[2\pi i \left(\frac{h}{2} + \frac{l}{2}\right)\right] + \exp\left[2\pi i \left(\frac{k}{2} + \frac{l}{2}\right)\right] \quad (8)$$

As shown before, we can see above if one of h, k, l is even and the other are odd (or vice versa) then we get $-1 \neq 1 - 1 - 1$ which is not allowed. However if all h, k, l are even or odd we obtain $-1 \neq 1 + 1 + 1$ which is true. Thus the (h,k,l) is always all odd or all even for the fcc.

2.4 Powder X-ray Diffraction

Transmission, fluorescence,

Xray diffraction, oscillating E field. When X ray hit the electrons the electrons start o vibrate in that frequency - Constructive and destructive interference

Bragg's law: $n\lambda = PD = 2s = 2d \sin \theta$

Crystal lattice: fcc, bcc

In powder diffraction we have all possible orientation sof the crystallites and some will be oriented in the right way for diffraction. Give rise to powder diffraction rings.

Scherrer's formula

$$t = \frac{k\lambda}{\beta \cos \theta} \quad (9)$$

Structure factor: Se photos. For a simple cubic we see all peaks, for bcc and fcc we loose some peaks.

3 Method

The energy of the x-rays are 17.45 keV.

The detector uses six semiconductors.

Calibrations of hex..., this is good since we have a lot of diffraction rings.

1. Put in all the data 2. mask out dead spots 3. Tell where the rings are 4. The image is converted to an intensity plot (Note that the x-axis is in 2θ).

Hot pixel, high peak with no width. The pixel can be burnt out.

Questions (we can remove this later but keep it now for keeping track)

1. Calculate the separations of the planes (111), (211), and (100) in a crystal in which the simple cubic unit cell has the side $a = 0.432$ nm. **DONE**
2. From Bragg's equation, what is the minimum d-spacing that can be observed with a wavelength of 0.07 nm? What do you need in order to observe smaller d spacings? **DONE**
3. Reflection from the (111) planes of a cubic crystal was observed at an incident angle $\theta = 11.2^\circ$ when Cu $K\alpha$ X-rays of wavelength 0.154 nm were used. What is the length of the side of the unit cell? **DONE**
4. Calcium carbonate crystals in the form of aragonite have an orthorhombic unit cell with dimensions $a = 0.574$ nm, $b = 0.796$ nm, $c = 0.495$ nm. Calculate the incident angles for the (100), (010), and (111) reflections using radiation of wavelength 0.083 nm. **DONE**
5. Calculate the allowed values of hkl for a body-centered cubic (bcc) and face-centered cubic (fcc) system that will give Bragg points on a diffraction pattern.
6. Find the two elements.
7. Measure the full width at half maximum (FWHM) of each peak of the two diffraction patterns and calculate the crystallite size using Scherrer's equation. Scherrer's equation is suitable for particle sizes below approximately 100 nm. Which property of light limits this kind of quantification?

- **Hint:** Constructive interference of two waves (and therefore diffraction) does not happen with any kind of light.

Bragg's law is based on the ideal case where a strictly monochromatic source is used. However, every X-ray source has a certain finite spectral bandwidth, $\frac{\Delta\lambda}{\lambda}$. As a result, the broader the spectral bandwidth, the broader the diffraction peaks.

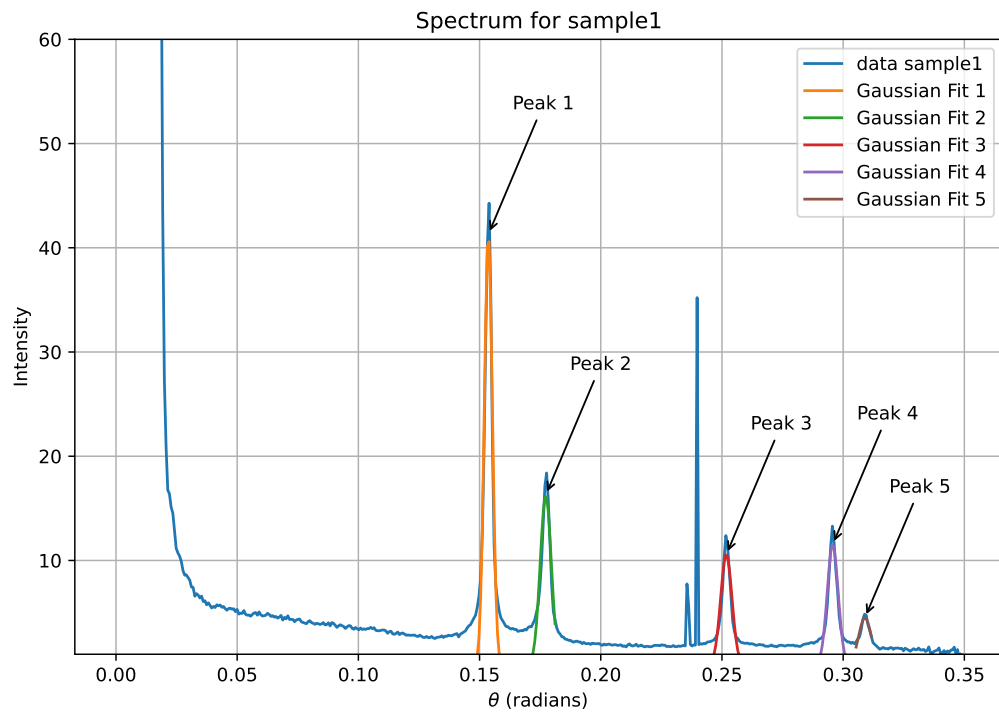
- (a) Prove that the spectral contribution of the source to the peak width, $\Delta\theta$, is related to the spectral bandwidth through the following relation:

$$\Delta\theta = \tan\theta \frac{\Delta\lambda}{\lambda} \quad (9.2)$$

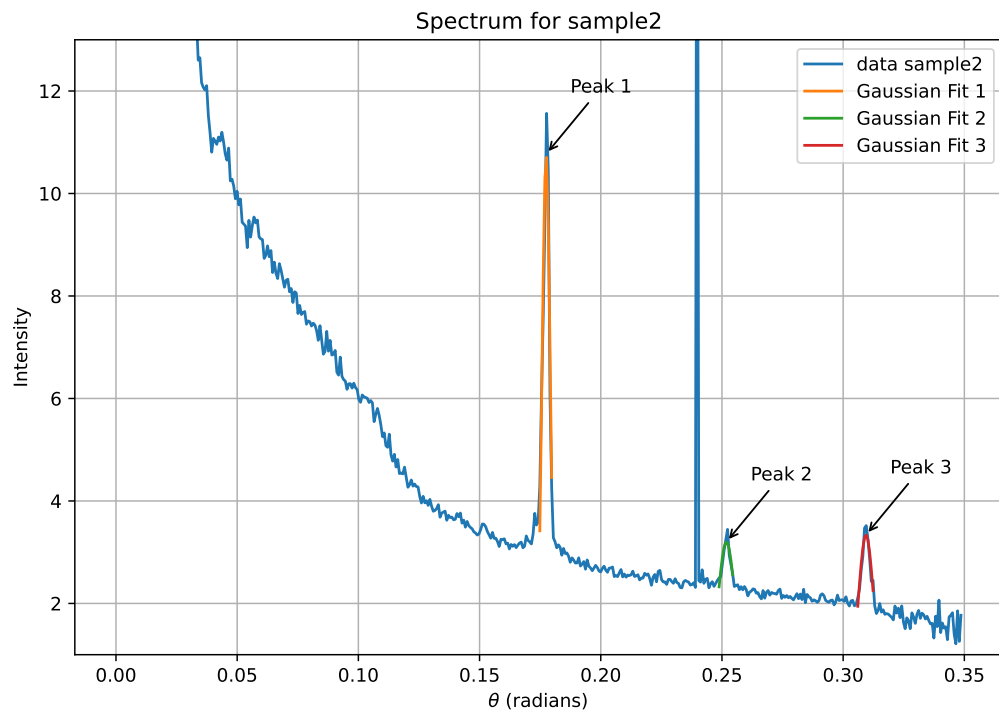
Hint: Differentiate Bragg's law.

- (b) Although this contribution affected the estimation you made in Exercise 7, it can be quantified to obtain a more exact crystallite size. Can you think of at least one other phenomenon or instrumental property that can affect the peak widths?

4 Result



(a) This is the first subfigure.



(b) This is the second subfigure.

Figure 1: This figure shows two subfigures with separate captions.

Table 1: Fitting results for Sample 1

Peak	Angle θ (radians)	Standard deviation σ (radians)	FWHM (radians)
Peak 1	$(0.153\,62 \pm 0.000\,10)$	$(0.001\,65 \pm 0.000\,10)$	$(0.003\,88 \pm 0.000\,23)$
Peak 2	$(0.177\,31 \pm 0.000\,19)$	$(0.002\,27 \pm 0.000\,20)$	$(0.005\,35 \pm 0.000\,46)$
Peak 3	$(0.251\,80 \pm 0.000\,18)$	$(0.002\,34 \pm 0.000\,18)$	$(0.005\,50 \pm 0.000\,43)$
Peak 4	$(0.295\,69 \pm 0.000\,15)$	$(0.002\,25 \pm 0.000\,15)$	$(0.005\,30 \pm 0.000\,35)$
Peak 5	$(0.309\,00 \pm 0.000\,15)$	$(0.002\,61 \pm 0.000\,21)$	$(0.006\,16 \pm 0.000\,49)$

Table 2: Fitting results for Sample 2

Peak	Angle θ (radians)	Standard deviation σ (radians)	FWHM (radians)
Peak 1	$(0.177\,46 \pm 0.000\,12)$	$(0.001\,72 \pm 0.000\,14)$	$(0.004\,04 \pm 0.000\,34)$
Peak 2	$(0.251\,86 \pm 0.000\,17)$	$(0.003\,80 \pm 0.000\,39)$	$(0.008\,95 \pm 0.000\,92)$
Peak 3	$(0.309\,42 \pm 0.000\,14)$	$(0.003\,26 \pm 0.000\,24)$	$(0.007\,68 \pm 0.000\,56)$

5 Discussion

6 Conclusion

References

- [1] Philip Hofmann. *Solid State Physics: An Introduction*. Wiley-VCH, 2nd edition, 2015.
- [2] Auden Ti and Hajar Jalili. *FYSC23: Powder X-ray Diffraction Laboratory Manual*. Lund, 2025. Lab instructors: Auden Ti (auden.ti_yun@sljus.lu.se) and Hajar Jalili (hajar.jalili@sljus.lu.se).