Laboratory Report

X Ray Diffraction

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Advanced Physics Laboratory Course (PL 701)

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Objectives:

1. To study the theory of X Ray diffraction, and use it on the given crystal (Lithium Fluoride)
2. To use different X-Rays (Copper and Molybdenum) and obtain the spectrum and hence the wavelength
3. To use a filter (Nickel for Copper and Zirconium for Molybdenum) and find out it's effects on the spectrum
4. To find out the structure of the given material (Copper).

Apparatus required

X Ray diffractometer, a crystal of Li-F, Cu and Mo metal targets, Computer for analysis and measuring.

Theory

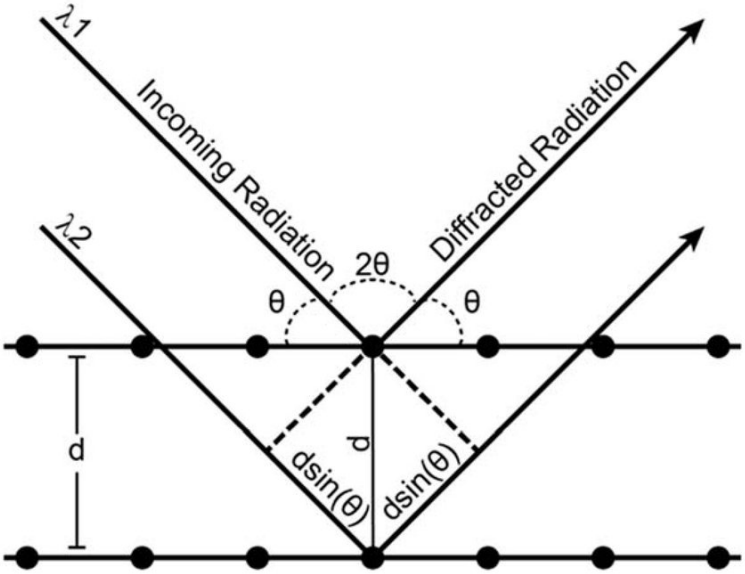
A strong method for figuring out a crystalline material's atomic and molecular structure is X-ray diffraction, which is employed in materials science and crystallography. X-rays experience diffraction when they come into contact with a crystalline material, producing a distinctive pattern of dispersed X-rays. Details regarding the arrangement of atoms or molecules within the crystal are contained in this pattern.
In most cases, X-rays produced by an X-ray tube are used for X-ray diffraction. An X-ray tube accelerates high-energy electrons before they crash with a metal target, such molybdenum or copper. These collisions cause X-rays to be released. Certain wavelengths of distinctive X-rays are generated, which are dictated by the target material's electron transitions. The energy of these X-rays is appropriate for diffraction experiments.
We mainly use the **Bragg's law of diffraction** which states

$$2dsin\theta = n\lambda$$

Where

- λ is the incident wavelength
- n is the order of the diffraction peak
- θ is the angle of incidence
- d is the spacing between the lattice planes

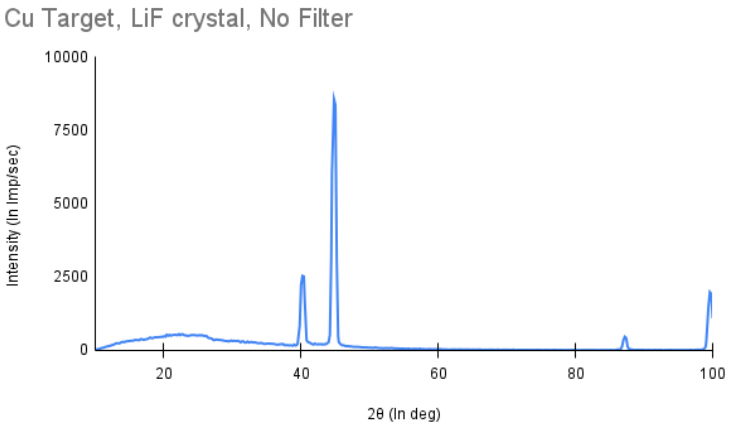
$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$



Observations

Target:Copper, Crystal:LiF, Filter: None

We obtain a spectrum like this



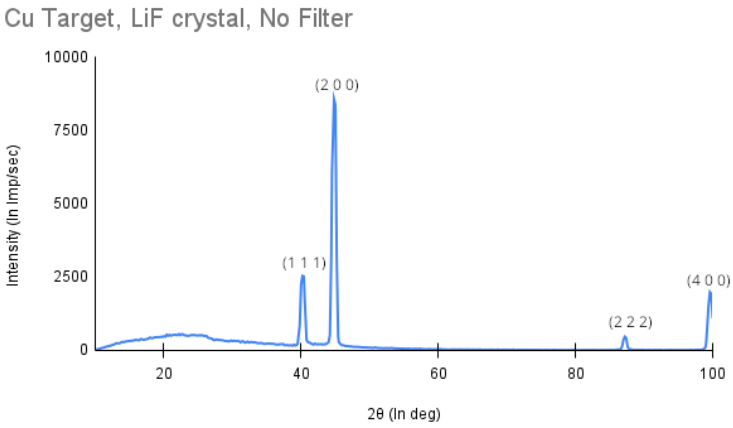
Spectrum of LiF XRD with Copper Target and no filter

We try to match it with the table given to us

2 θ	Intensity	h	k	l
38.696	95	1	1	1
44.996	100	2	0	0
65.494	48	2	2	0
78.765	10	3	1	1
82.998	11	2	2	2
99.628	3	4	0	0
112.967	4	3	3	1
117.606	14	4	2	0
139.134	13	4	2	2

2θ vs Intensity data for Lithium Fluoride

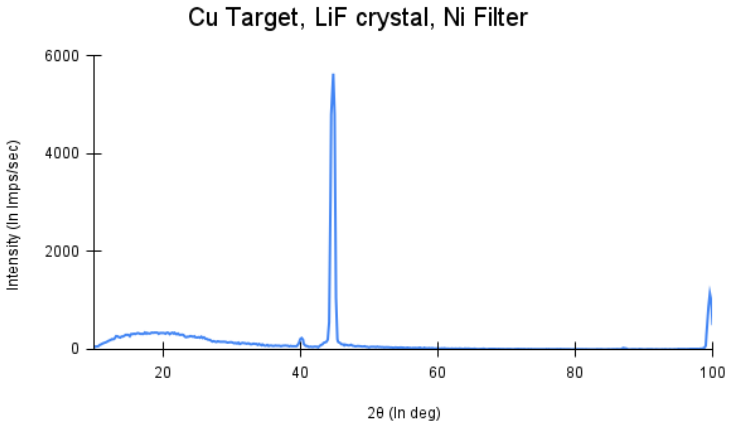
At the first thought, it seems like the 38.696, 44.996, to an extent 82.998 and 99.628 bands are visible. So we mark that.



Marked spectrum of LiF XRD with Copper Target and no filter

Target:Copper, Crystal:LiF, Filter:Ni

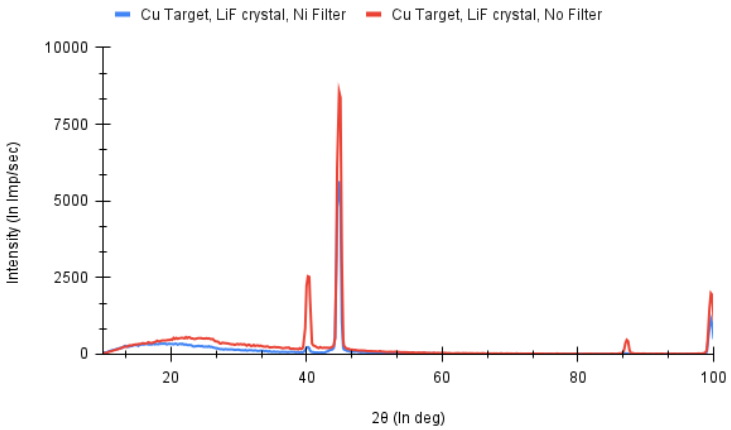
We obtain a spectrum like this



Spectrum of LiF XRD with Copper Target and Ni filter

This comes as a surprise for us as

1. There is no (1 1 1) band or (2 2 2) band in the filtered one
2. There are bands of only one kind predominant, that is the (h,0,0) type bands. We present the combined two graphs together for a better inference



Combined spectrum of LiF XRD and Copper Target, with and without filter

Inferences

- The presence of just (h,0,0) bands should imply the crystal lattice is oriented in a single direction, hence it is a mono-crystalline solid.
- We can find out the wavelength of the X-Ray with this spectrum. We know

$$2dsin\theta = n\lambda$$

We have

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

We have a to be 402 pm or 4.02 Å which is the lattice constant of LiF. Calculating the λ of the X-Ray, we get

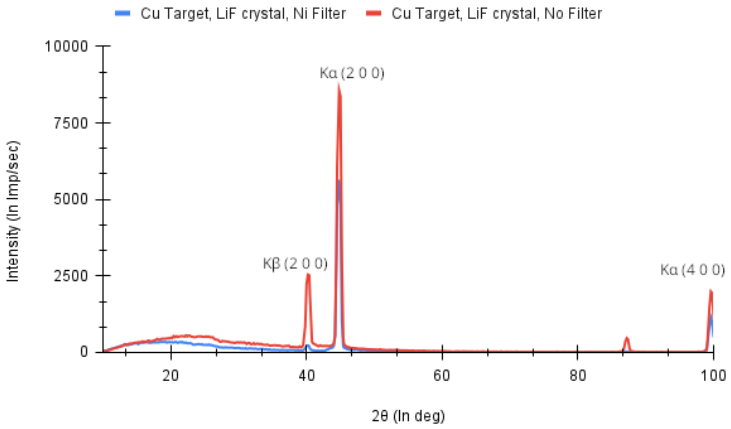
$$\lambda = \frac{2 \times 4.02 \times 10^{-10}}{\sqrt{2^2 + 0^2 + 0^2}} sin(44.8/2)$$
$$\lambda = 1.531\mathring{A}$$

The uncertainty stems up from the least count of the machine (0.2 degrees). Accounting that
$\lambda = \mathbf{1.531 \pm 0.007\ \mathring{A}}$

- So the line which we incorrectly marked as (1 1 1) was actually the K_β line, the major one being the K_α line. Doing the similar calculations for the K_β line, we get

$$\lambda = \frac{2 \times 4.02 \times 10^{-10}}{\sqrt{2^2 + 0^2 + 0^2}} sin(40.2/2)$$
$$\lambda = 1.381 \mathring{A}$$

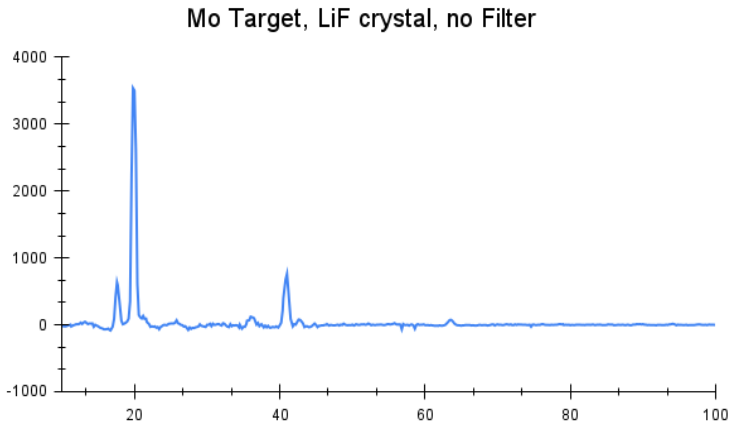
With uncertainty, $\lambda = \mathbf{1.381 \pm 0.007}$ **Å** Now finally marking both the spectrum with the correct markings



- The role of the filter is that it shunts away the K_β line, hence we get only the K_α spectrum.

Target:Molybdenum, Crystal:LiF, Filter: None

We obtain a spectrum like this



Spectrum of LiF XRD with Molybdenum Target and no filter

We try to match it with the table given to us

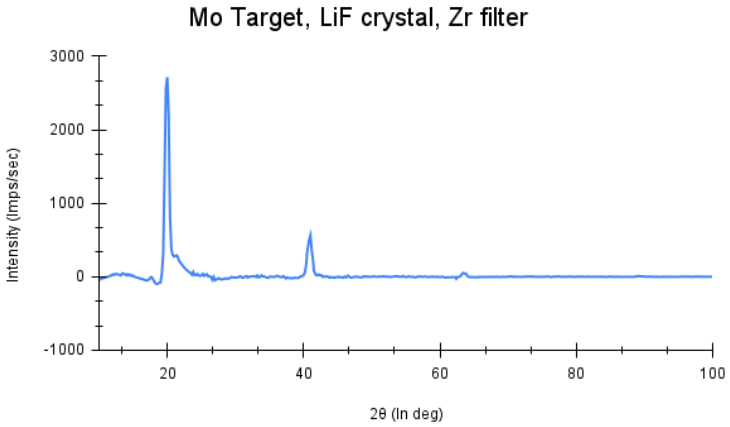
2 θ	Intensity	h	k	l
17.548	95	1	1	1
20.295	100	2	0	0
28.843	48	2	2	0
33.971	10	3	1	1
35.525	11	2	2	2
41.251	3	4	0	0
45.146	4	3	3	1
46.387	14	4	2	0
51.119	13	4	2	2

2θ vs Intensity data for Lithium Fluoride

Similar to case with Copper, it seems like there is a (1 1 1) band. But we shall not mark it this time, being a bit more cautious.

Target:Molybdenum, Crystal:LiF, Filter:Ni

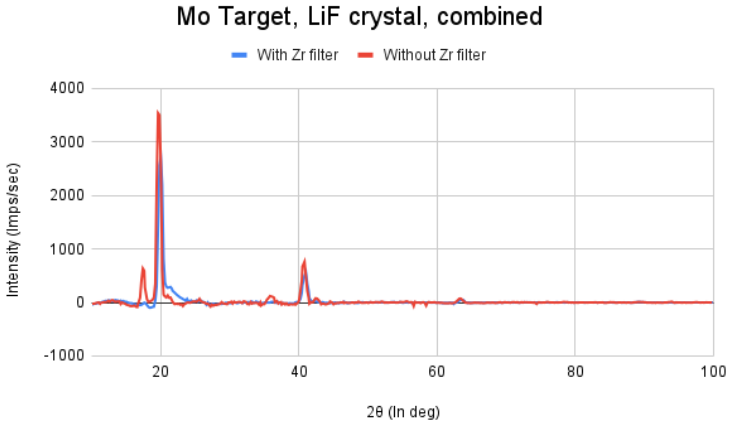
We obtain a spectrum like this



Spectrum of LiF XRD with Copper Target and Ni filter

This was expected this time, to nail the points once more

1. There is no (1 1 1) band or (2 2 2) band in the filtered one
2. There are bands of only one kind predominant, that is the (h,0,0) type bands. We present the combined two graphs together for a better inference



Combined spectrum of LiF XRD and Copper Target, with and without filter

We do the same analysis once more

Inferences

- The presence of just (h,0,0) bands should imply the crystal lattice is oriented in a single direction, hence it is a mono-crystalline solid.
- We can find out the wavelength of the X-Ray with this spectrum. We know

$$2dsin\theta = n\lambda$$

We have

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

We have a to be 402 pm or 4.02 Å which is the lattice constant of LiF. Calculating the λ of the X-Ray, we get

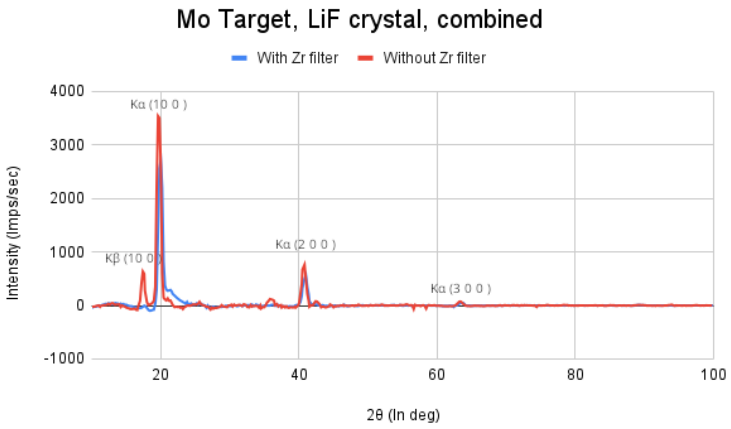
$$\lambda = \frac{2 \times 4.02 \times 10^{-10}}{\sqrt{2^2 + 0^2 + 0^2}} sin(20/2)$$
$$\lambda = 0.698 \mathring{A}$$

The uncertainty stems up from the least count of the machine (0.2 degrees). Accounting that
$\lambda = \mathbf{0.698 \pm 0.007}$ **Å**

- So the line which we incorrectly marked as (1 1 1) was actually the K_β line, the major one being the K_α line. Doing the similar calculations for the K_β line, we get

$$\lambda = \frac{2 \times 4.02 \times 10^{-10}}{\sqrt{2^2 + 0^2 + 0^2}} sin(17.6/2)$$
$$\lambda = 0.615 \mathring{A}$$

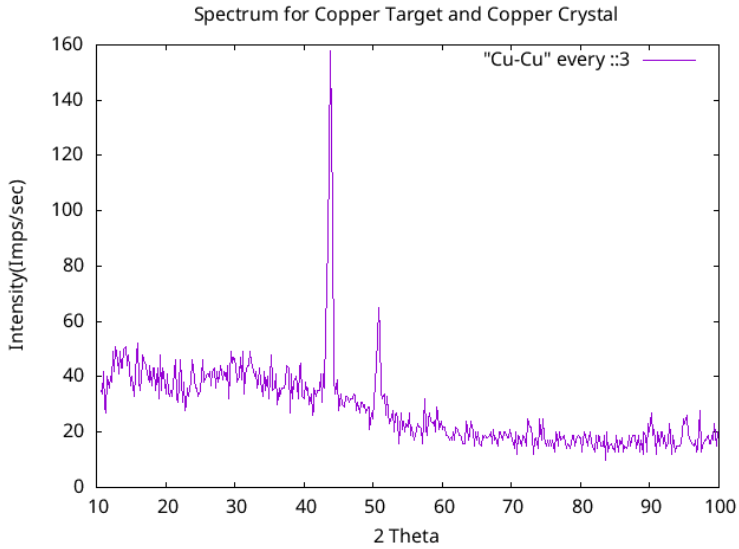
With uncertainty, $\lambda = \mathbf{0.615 \pm 0.007}$ **Å** Now finally marking both the spectrum with the correct markings



- The role of the filter is that it shunts away the K_β line, hence we get only the K_α spectrum.

Target:Copper, Crystal:Cu, Filter: Ni

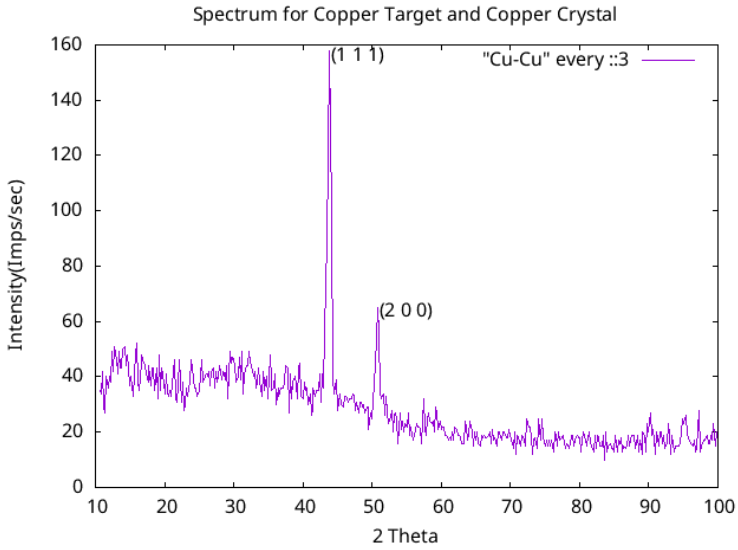
Now we know the wavelength of the X-Ray (1.531 $\pm$ 0.007 Å) when a copper target is used, we now try to find the structure of Copper using this. We also use this as an opportunity to switch the plotting software to GNUplot.



We refer the table for Copper

2θ	Intensity	h	k	l
43.297	100	1	1	1
50.433	46	2	0	0
74.130	20	2	2	0
89.931	17	3	1	1
95.139	5	2	2	2
116.919	3	4	0	0
136.507	9	3	3	1
144.714	8	4	2	0

Now we try labelling the peaks, there is a peak at 43.8, 50.8. The other peaks are shadowed by noise.



Since there are two different planes present, this is a polycrystalline sample. Now we try to find the lattice parameter.

$$2dsin\theta = n\lambda$$

We have

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

So we can rearrange as

$$d = \frac{n\lambda}{2sin\theta}$$

$$a = \frac{\lambda}{2sin\theta} \times \sqrt{h^2 + k^2 + l^2}$$

Plugging in $\lambda = 1.531\mathring{A}, \theta = 21.9°, \sqrt{h^2 + k^2 + l^2} = \sqrt{3}$.

$$a = 3.554\mathring{A}$$

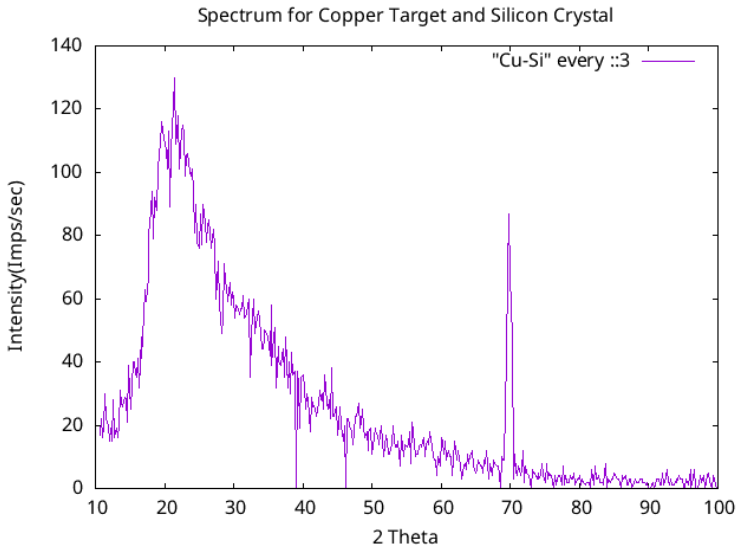
Plugging in $\lambda = 1.531\mathring{A}, \theta = 25.4°, \sqrt{h^2 + k^2 + l^2} = \sqrt{4}$

$$a = 3.569\mathring{A}$$

Error obtained = $\pm$ 0.003Å

Target:Copper, Crystal:Si, Filter: Ni

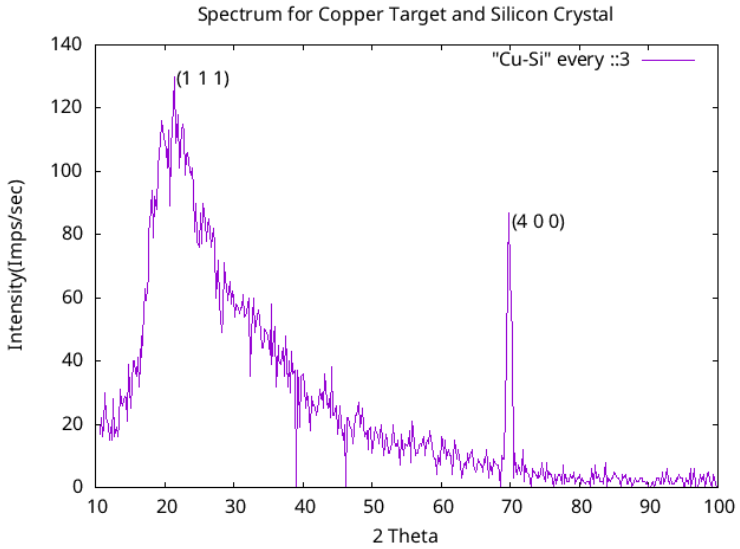
Next we try to analyse the Silicon crystal given to us.



At the first sight, the right side of spectrum is noisy, suggesting contamination. Which was suspected as the Silicon crystal looked dirty. Looking at the database, we have peaks visible at 27.8 and 69.8. All the other potential peaks are subsumed by noise and impurities.

2θ	Intensity	h	k	l
28.443	100	1	1	1
47.304	55	2	2	0
56.122	30	3	1	0
69.132	6	4	0	0
76.379	11	3	3	1
88.029	12	4	2	2
94.957	6	5	1	1

Si database The higher values of 2θ are excluded due to instrument measuring only till $2\theta = 100$



Since there are two different planes present, this is a polycrystalline solid.Now we try to find the lattice parameter.

$$2dsin\theta = n\lambda$$

We have

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

So we can rearrange as

$$d = \frac{n\lambda}{2sin\theta}$$

$$a = \frac{\lambda}{2sin\theta} \times \sqrt{h^2 + k^2 + l^2}$$

Plugging in $\lambda = 1.531\mathring{A}, \theta = 13.9, \sqrt{h^2 + k^2 + l^2} = \sqrt{3}$.

$$a = 5.519\mathring{A}$$

Plugging in $\lambda = 1.531\mathring{A}, \theta = 34.9, \sqrt{h^2 + k^2 + l^2} = \sqrt{16}$

$$a = 5.35\mathring{A}$$

Error obtained = ± 0.003Å

Result

1. The X-Ray wavelength of Copper target is
   - K_α**= 1.531 ± 0.007 Å**
   - K_β**= 1.381 ± 0.007 Å**
2. The X-Ray wavelength of Molybdenum target is
   - K_α**= 0.698 ± 0.007 Å**
   - K_β**= 0.615 ± 0.007 Å**
3. We analysed a Copper crystal (Supposedly FCC)
   (a) Lattice parameter for (h h h) plane = **= 3.554 ± 0.003 Å**
   (b) Lattice parameter for (h 0 0) plane = **= 3.569 ± 0.003 Å**
4. We analysed an impure Silicon crystal (Supposedly Diamond)
   (a) Lattice parameter for (h h h) plane = **= 5.519 ± 0.003 Å**
   (b) Lattice parameter for (h 0 0) plane = **= 5.351 ± 0.003 Å**