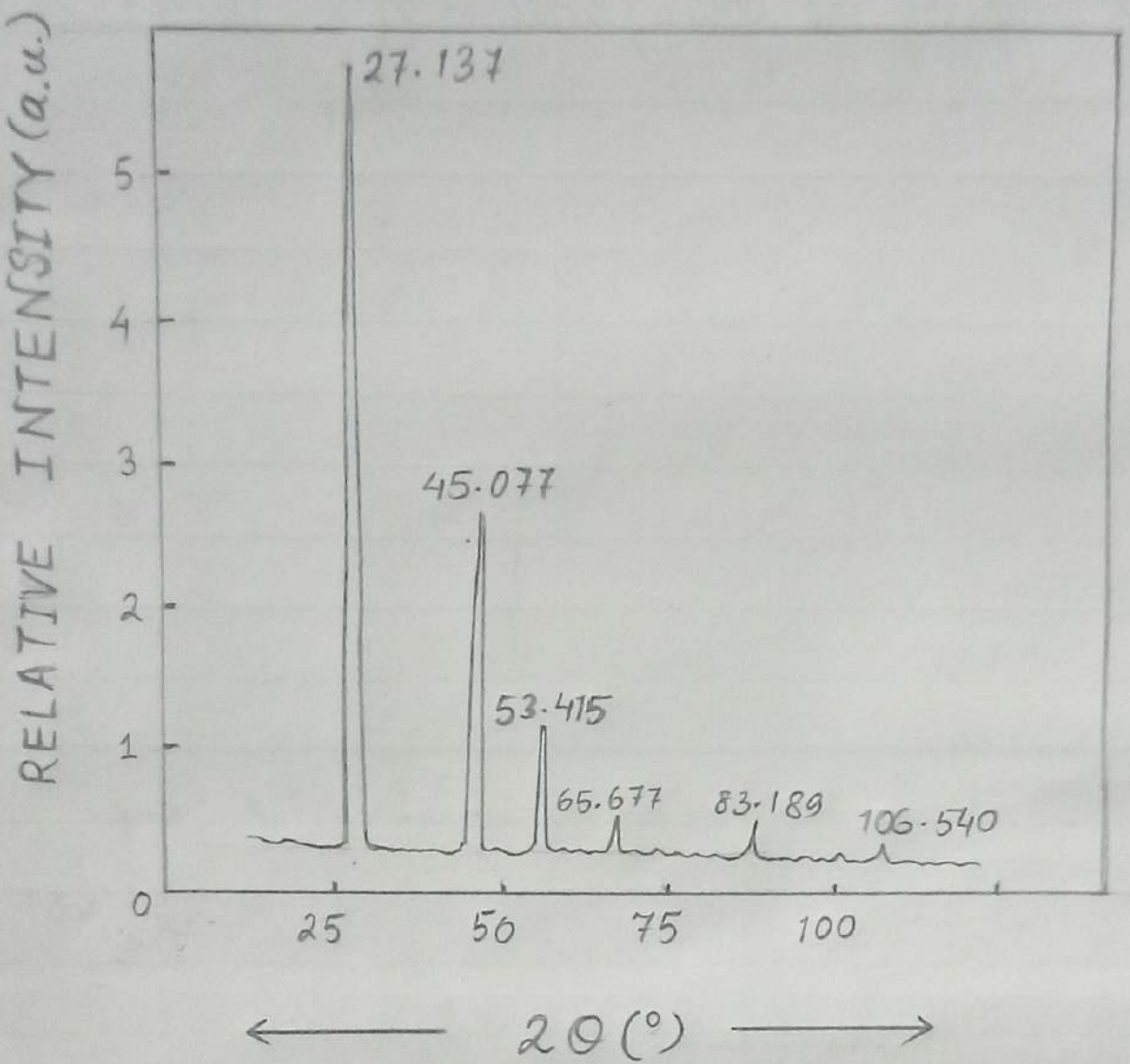
RELATIVE INTENSITY (a.u.)
27.137
45.077
53.415
65.677
83.189
106.540
0
1
2
3
4
5
25
50
75
100
2Θ (°)

XRD pattern

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Calculation of Lattice Cell Parameters – X-ray Diffraction

AIM : To calculate the lattice cell parameters from the powder X-ray diffraction data.

APPARATUS : Powder X-ray diffraction diagram.

FORMULA :

For cubic crystal : $1/d^2 = (h^2+k^2+l^2)/a^2$

For tetragonal crystal : $1/d^2 = \{(h^2+k^2)/a^2 + l^2/c^2\}$

For orthorhombic crystal : $1/d^2 = \{(h^2/a^2) + (k^2/b^2) + (l^2/c^2)\}$

The lattice parameter and interplanar distance for a cubic crystal are :

$$a = (\lambda/2\sin\theta)(\sqrt{h^2+k^2+l^2})\ \text{Å}$$

$$d = \left(a/\sqrt{h^2+k^2+l^2}\right)\ \text{Å}$$

where : a = lattice parameter
d = Interplanar distance.
λ = Wavelength of CuKα radiation (1.5405 Å)
h, k, l = Miller integers.

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PRINCIPLE : Braggs law is the theoretical basis for X-ray diffraction.

$$(\sin^2\theta)_{hkl} = (\lambda^2/4a^2)(h^2 + k^2 + l^2)$$

Each of the Miller indices can take values 0, 1, 2, 3, ... Thus, the factor $(h^2 + k^2 + l^2)$ takes the values given in Table aside.

PROCEDURE : From the 2θ values on a powder photograph, the θ values are obtained. The $\sin^2\theta$ values are tabulated. From that values of $[1 \times (\sin^2\theta/\sin^2\theta_{min})]$, $[2 \times (\sin^2\theta/\sin^2\theta_{min})]$ and $[3 \times (\sin^2\theta/\sin^2\theta_{min})]$ are determined and tabulated. The values of $[3 \times (\sin^2\theta/\sin^2\theta_{min})]$ are rounded to the nearest integer. This gives the value of $h^2 + k^2 + l^2$. From these the values of h, k, l are determined.

OBSERVATION :

Value of $h^2+k^2+l^2$ for different planes

	h, k, l	$h^2+k^2+l^2$	h, k, l	$h^2+k^2+l^2$
1.	100	1	300	9
2.	110	2	310	10
3.	111	3	311	11
4.	200	4	322	12
5.	210	5	320	13
6.	211	6	321	14
7.	220	8	400	16
8.	221	9	410	17

	2θ	θ	$\sin^2\theta$	$1\times\frac{\sin^2\theta}{\sin^2\theta_{min}}$	$2\times\frac{\sin^2\theta}{\sin^2\theta_{min}}$	$3\times\frac{\sin^2\theta}{\sin^2\theta_{min}}$	$h^2+k^2+l^2$	hkl	a Å	d Å
1.	27.137	13.569	0.0550	1	2	3	3	111	5.6863	3.2830
2.	45.077	22.539	0.1469	2.6714	5.3428	8.0142 ≈ 8	8	220	5.6836	2.0095
3.	53.415	26.708	0.2020	3.6727	7.3454	11.0181 ≈ 11	11	311	5.6839	1.7138
4.	65.677	32.839	0.2941	5.3473	10.6946	16.0419 ≈ 16	16	400	5.6816	1.4204
5.	83.189	41.595	0.4407	8.0127	16.0254	24.0381 ≈ 24	24	422	5.6841	1.1603
6.	106.54	53.27	0.6423	11.6782	23.3563	35.0346 ≈ 35	35	531	5.6857	0.9611
								Mean :	5.6842	1.7580

CALCULATION :

① $2\theta = 27.137$	$\frac{2 \times \sin^2\theta}{\sin^2\theta_{min}} = 2 \times \frac{0.055}{0.055} = 2$	$a = (\lambda / 2\sin\theta)\sqrt{h^2+k^2+l^2}$
$\therefore \theta = 27.137/2 = 13.569$		$= (1.5405/2 \times 0.2346)\sqrt{3}$
$\sin\theta = \sin 13.569 = 0.2346$	$\frac{3 \times \sin^2\theta}{\sin^2\theta_{min}} = \frac{3 \times 0.055}{0.055} = 3$	$= 5.6863$ Å
$\sin^2\theta = (0.2346)^2 = 0.0550$		
$\sin^2\theta_{min} = 0.0550$	$h^2+k^2+l^2 = 3$	$d = (a/\sqrt{h^2+k^2+l^2})$
	$h\ k\ l = 1\ 1\ 1$	$= 5.6863/\sqrt{3}$
$\frac{1 \times \sin^2\theta}{\sin^2\theta_{min}} = 1 \times \frac{0.055}{0.055} = 1$		$= 3.2830$ Å
	λ (given) $= 1.5405$ Å	

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② $2\theta = 45.077$

$\therefore \theta = 45.077/2 = 22.53$

$\sin\theta = \sin 22.5385 = 0.3833$

$\sin^2\theta = (0.3833)^2 = 0.1469$

$\sin^2\theta_{min} = 0.0550$

$1 \times \frac{\sin^2\theta}{\sin^2\theta_{min}} = 1 \times \frac{0.1469}{0.0550} = 2.6714$

$2 \times \frac{\sin^2\theta}{\sin^2\theta_{min}} = 2 \times \frac{0.1469}{0.0550} = 5.3428$

$3 \times \frac{\sin^2\theta}{\sin^2\theta_{min}} = 3 \times \frac{0.1469}{0.0550} = 8.0142$

$h^2+k^2+l^2 = 8$

$hkl = 220$

$\lambda(given) = 1.5405$ Å

$a = (\lambda/2\sin\theta)(\sqrt{h^2+k^2+l^2})$

$= (1.5405/2\sin 22.5385)\sqrt{8}$

$= 5.6836$ Å

$d = (a/\sqrt{h^2+k^2+l^2})$

$= (5.6836/\sqrt{8})$

$= 2.0095$ Å

---

③ $2\theta = 53.415$

$\therefore \theta = 53.415/2 = 26.708$

$\sin\theta = \sin 26.708 = 0.4494$

$\sin^2\theta = (0.4494)^2 = 0.2020$

$\sin^2\theta_{min} = 0.0550$

$1 \times \frac{\sin^2\theta}{\sin^2\theta_{min}} = 1 \times \frac{0.2020}{0.055} = 3.6727$

$2 \times \frac{\sin^2\theta}{\sin^2\theta_{min}} = 2 \times \frac{0.2020}{0.055} = 7.345$

$3 \times \frac{\sin^2\theta}{\sin^2\theta_{min}} = 3 \times \frac{0.2020}{0.055} = 11.0181$

$h^2+k^2+l^2 = 11$

$hkl = 311$

$\lambda(given) = 1.5405$ Å

$a = (\lambda/2\sin\theta)\sqrt{h^2+k^2+l^2}$

$= (1.5405/2\times 0.4494)\sqrt{11}$

$= 5.6839$ Å

$d = (a/\sqrt{h^2+k^2+l^2})$

$= (5.6839/\sqrt{11})$

$= 1.7138$ Å

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④ $2\theta = 65.677$

$\therefore \theta = 65.677/2 = 32.839$

$\sin\theta = \sin 32.839 = 0.5423$

$\sin^2\theta = (0.5423)^2 = 0.2941$

$\sin^2\theta_{min} = 0.0550$

$1 \times \frac{\sin^2\theta}{\sin^2\theta_{min}} = 1 \times \frac{0.2941}{0.055} = 5.3473$

$2 \times \frac{\sin^2\theta}{\sin^2\theta_{min}} = 2 \times \frac{0.2941}{0.055} = 10.69$

$3 \times \frac{\sin^2\theta}{\sin^2\theta_{min}} = 3 \times \frac{0.2941}{0.055} = 16.04$

$h^2+k^2+l^2 = 16$

$hkl = 400$

$\lambda(given) = 1.5405$ Å

$a = (\lambda/2\sin\theta)\sqrt{h^2+k^2+l^2}$

$= (1.5405/2\times 0.5423)\sqrt{16}$

$= 5.6816$

$d = (a/\sqrt{h^2+k^2+l^2})$

$= 5.6816/\sqrt{16}$

$= 1.4204$ Å

⑤ $2\theta = 83.189$ $\therefore \theta = 83.189/2 = 41.595$ $\sin\theta = \sin 41.595 = 0.6639$ $\sin^2\theta = (0.6639)^2 = 0.4407$ $\sin^2\theta_{min} = 0.055$ $1 \times \frac{\sin^2\theta}{\sin^2\theta_{min}} = 1 \times \frac{0.4407}{0.055} = 8.012$	$2 \times \frac{\sin^2\theta}{\sin^2\theta_{min}} = 2 \times \frac{0.4407}{0.055} = 16.025$ $3 \times \frac{\sin^2\theta}{\sin^2\theta_{min}} = 3 \times \frac{0.4407}{0.055} = 24.038$ $h^2+k^2+l^2 = 24$ $hkl = 422$ $\lambda(\text{given}) = 1.5404\ \text{Å}$	$a = (\lambda/2\sin\theta)\sqrt{h^2+k^2+l^2}$ $= (1.5404/2\sin 41.595)\sqrt{24}$ $= 5.6857\ \text{Å}$ $d = a/\sqrt{h^2+k^2+l^2}$ $= 5.6857/\sqrt{24}$ $= 1.1603\ \text{Å}$
⑥ $2\theta = 106.54$ $\therefore \theta = 106.54/2 = 53.27$ $\sin\theta = \sin 53.27 = 0.8015$ $\sin^2\theta = (0.8015)^2 = 0.6423$ $\sin^2\theta_{min} = 0.055$ $1 \times \frac{\sin^2\theta}{\sin^2\theta_{min}} = 1 \times \frac{0.6423}{0.055}$ $= 11.6782$	$2 \times \frac{\sin^2\theta}{\sin^2\theta_{min}} = 2 \times \frac{0.6423}{0.055} = 23.36$ $3 \times \frac{\sin^2\theta}{\sin^2\theta_{min}} = 3 \times \frac{0.6423}{0.055} = 35.03$ $h^2+k^2+l^2 = 35$ $hkl = 531$ $\lambda(\text{given}) = 1.5404\ \text{Å}$	$a = (\lambda/2\sin\theta)\sqrt{h^2+k^2+l^2}$ $= (1.5404/2\times 0.8015)\sqrt{35}$ $= 5.6857\ \text{Å}$ $d = a/\sqrt{h^2+k^2+l^2}$ $= 5.6857/\sqrt{35}$ $= 0.9611\ \text{Å}$

Average of Lattice parameter (a) = $(5.6863 + 5.6836 + 5.6839 + 5.6816 + 5.6841 + 5.6857)/6$

$= 5.6842\ \text{Å}$

Average of Interplanar distance (d) = $(3.2830 + 2.0095 + 1.7138 + 1.4204 + 1.1603 + 0.9611)/6$

$= 1.7580\ \text{Å}$

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LATTICE DETERMINATION :

Lattice Type	Rule for reflection to be observed.
Primitive P	None
Body Centred I	$h k l : h+k+l = 2n$
Face centred F	h k l : h, k, l either all odd or all even.

Depending on the nature of the h, k, l values, the lattice type can be determined.

RESULT :

The lattice cell parameters are calculated theoretically from the powder X-ray diffraction pattern and the values are tabulated as;

Average lattice parameter (a) = 5.6842 Å

Average of Interplanar distance (d) = 1.7580 Å

—x—