X-ray Diffraction Analysis

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Extinction/Selection Rules

Even if Bragg's law get satisfied, some reflections go missing

Bravais Lattice	Reflections which *may be* present	Reflections necessarily absent
Simple	all	None
Body centred	(h + k + l) even	(h + k + l) odd
Face centred	h, k and l unmixed *(i.e. all even or all odd)*	h, k and l mixed
Diamond Cubic	Either, ❶ h, k and l are all odd *or* ❷ all are even & *(h + k + l) divisible by 4*	h, k and l are mixed

How to find out crystal structure

$$\lambda = 2d\sin\theta$$

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

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

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

$$h^2 + k^2 + l^2 \prec \sin^2\theta$$

Allowed reflections in SC, FCC, BCC & DC crystals

$h^2 + k^2 + l^2$	SC	FCC	BCC	DC
1	100			
2	110		110	
3	111	111		111
4	200	200	200	
5	210			
6	211		211	
7				
8	220	220	220	220
9	300, 221			
10	310		310	
11	311	311		311
12	222	222	222	
13	320			
14	321		321	
15				
16	400	400	400	400
17	410, 322			
18	411, 330		411, 330	
19	331	331		331

Cannot be expressed as $(h^2+k^2+l^2)$

Extinction/Selection Rules

The ratio of $(h^2 + k^2 + l^2)$ derived from extinction rules

SC	1	2	3	4	5	6	8	…
BCC	1	2	3	4	5	6	7	…
FCC	3	4	8	11	12	…		
DC	3	8	11	16	…			

- Note that we have to consider the ratio of only two lines to distinguish FCC and DC. I.e. if the ratios are 3:4 then the lattice is FCC.

- But, to distinguish between SC and BCC we have to go to 7 lines!

How to find out crystal structure

1. Start with 2θ values and generate a set of $Sin^2\theta$ values
2. Normalize the $Sin^2\theta$ by generating $Sin^2\theta_n / Sin^2\theta_1$
3. Clear fractions from normalized column
4. Speculate on hkl values that if expressed as $(h^2+k^2+l^2)$, it will generate the sequence of the clear fraction column

Example -1

No need to multiply as it is simple cubic structure

2θ	θ	$Sin\theta$	$Sin^2\theta$	$Sin^2\theta_n/Sin^2\theta_1$	Multiplicity factor	n	(hkl)
25.45	12.73	0.2203	0.0485	1.0	1 (not needed)	1.0	
36.30	18.15	0.3115	0.0970	2.0		2.0	
44.86	22.43	0.3816	0.1456	3.0		3.0	
52.28	26.14	0.4406	0.1941	4.0		4.0	
59.02	29.51	0.4926	0.2426	5.0		5.0	
65.31	32.66	0.5396	0.2911	6.0		6.0	
77.08	38.54	0.6231	0.3882	8.0		8.0	
82.73	41.37	0.6609	0.4367	9.0		9.0	

Example -2

- 7 is not at all possible, so multiply by 2

2θ	θ	Sinθ	$Sin^2θ$	$Sin^2θ_n/Sin^2θ_1$	Multiplicity factor	n	(hkl)
38.60	19.30	0.33051	0.1092	1.0	2	2.0	
55.71	27.86	0.46724	0.2183	2.0		4.0	
69.70	34.85	0.57143	0.3265	3.0		6.0	
82.55	41.28	0.65967	0.4352	4.0		8.0	
95.00	47.50	0.73728	0.5436	5.0		10.0	
107.67	53.84	0.80732	0.6518	6.0		11.9	
122.03	61.02	0.87475	0.7652	7.0		14.0	

Multiplicity factor is the number to convert $Sin^2θ_n/Sin^2θ_1$ ratio to integer values

Example -3

2θ	θ	$Sin\theta$	$Sin^2\theta$	$Sin^2\theta_n/Sin^2\theta_1$	Multiplicity factor	n	(hkl)
44.86	22.43	0.3816	0.1456	1.0	3	3.0	
52.28	26.14	0.4406	0.1941	1.3		4.0	
77.08	38.54	0.6231	0.3882	2.7		8.0	
93.87	46.94	0.7306	0.5337	3.7		11.0	
99.48	49.74	0.7631	0.5824	4.0		12.0	

Multiplicity factor is the number to convert $Sin^2\theta_n/Sin^2\theta_1$ ratio to integer values

Summary

Assignments

1. From an X-Ray powder diffraction of a pure element, peaks at the following 2θ values in degrees were obtained 38.7, 45.4, 65.7, 78.8, 83.0, 99.6, 112.5, 117.0, 138.1, and 164.2. Copper K_α radiation was used. Find the lattice parameter and the crystal structure.
2. A BCC crystal is used to measure the wavelength of some X-rays. The Bragg angle for reflection from (110) plane is 20.2°. What is the wavelength? The lattice parameter of the crystal is 3.15 Å.
3. Determine the Miller indices of cubic crystal plane that intersects the position coordinates (1,1/4, 0), (1,1,1/2), and (3/4,1,1/4).
4. NaCl has the FCC lattice with a = 5.63 Å. What is the spacing of {100} plane?
5. In powder diffraction pattern for lead with radiation of $\lambda = 1.54$ Å the (220) Bragg reflection angle is $\theta = 32^\circ$. What is the radius of atom?