MLL100: Introduction to Materials Science and Engineering – Lab Session

Student Names: Entry Nos: Group No:

Experiment 3: X-ray Diffraction Studies

Reading reference: Section 3.6 (pp.42-45) in Material Science and Engineering book (5th Edn.) by V. Raghavan.

0.33 marks each from row 4 to 27. No partial marking **(8 Marks)**

Q1: Make a list of *h k l* values for increasing values of $h^2 + k^2 + l^2$ (note that for some values of $h^2 + k^2 + l^2$ there is no corresponding *h k l* whereas for some there are more than one set of *h k l* values). Then using the extinction rules for cubic crystals **(Table 3.3 of pg. 44 in your textbook**) indicate with a tick the reflections that are present in SC, BCC, FCC and DC crystals. Thus determine the ratios of $h^2 + k^2 + l^2$ values for the first few peaks from these crystals.

$h^2 + k^2 + l^2$	*h k l*	$h^2 + k^2 + l^2$ value for allowed reflection			
		SC	BCC	FCC	DC
1	100	1			
2	110	2	2		
3	111	3		3	3
4	200	4	4	4	
5	210	5			
6	211	6	6		
7					
8	220	8	8	8	8
9	300,221	9			
10	310	10	10		
11	311	11		11	11
12	222	12	12	12	
13	320	13			
14	321	14	14		
15					
16	400	16	16	16	16
17	410,322	17			
18	411,330	18	18		
19	331	19		19	19
20	420	20	20	20	
21	421	21			
22	332	22	22		
23					

24	422	24	24	24	24
25	500,430	25			
26	510,431	26	26		
27	511,333	27		27	27

Q2: X-ray diffraction pattern from a cubic crystal system (12 marks)

You are provided with an x-ray diffraction pattern from a cubic crystal (SC/BCC/FCC/DC). You should index the peaks and determine the Bravais lattice. Place the indices determined by you next to the peaks on the diffraction pattern.

Determine the lattice parameter, *a,* using the data for the highest diffraction angle. Use the value of λ =1.54 Å.

For following method, i.e., 2theta, theta, sin2theta etc, you give 5 marks
Next look at h2+k2+l2 values and award 4 marks
Correct cyrstal structure: 1 mark
Finally, for correct lattice parameter or approximate value, award 2 marks
In this case, (h square+k square +l square) =9 number is missing

Set No: A

Peaks	$2\theta_i$	θ_i	$Sin^2(\theta_i)$	$\frac{Sin^2(\theta_i)}{Sin^2(\theta_1)}$	Clear Fraction/Integer approx.	$h^2+k^2+l^2$	hkl
1	23	11.5	0.039	1	1	1	100
2	32	16	0.076	1.91	2	2	110
3	40	20	0.117	2.94	3	3	111
4	46	23	0.153	3.84	4	4	200
5	53	26.5	0.199	5	5	5	210
6	58	29	0.235	5.91	6	6	211
7	68	34	0.313	7.87	8	8	220
~~8~~	~~72~~	~~36~~	~~0.345~~	~~8.69~~	~~9~~	~~9~~	~~221~~
9	77	38.5	0.388	9.75	10	10	310
10	82	41	0.430	10.83	11	11	311
11	87	43.5	0.473	11.92	12	12	222

Bravais Lattice: cP

Primitive Cubic Crystal, a=3.88 Å

For following method, i.e., 2theta, theta, sin2theta etc, you give 5 marks
Next look at h2+k2+l2 values and award 4 marks
Correct cyrstal structure: 1 mark
Finally, for correct lattice parameter or approximate value, award 2 marks

Set No: B

Peaks	$2\theta_i$	θ_i	$Sin^2(\theta_i)$	$\frac{Sin^2(\theta_i)}{Sin^2(\theta_1)}$	Clear Fraction	$h^2+k^2+l^2$	*hkl*
1	28.45	14.23	0.061	1	3	3	111
2	47.32	23.66	0.161	2.67	8	8	220
3	56.14	28.07	0.221	3.67	11	11	311
4	69.15	34.58	0.322	5.33	15.99	16	400
5	76.4	38.2	0.382	6.33	19	19	331
6	88	44	0.483	7.99	23.97	24	422

Bravais Lattice: cF

Diamond Cubic Crystal, a=5.430 Å

Set No: C

Peaks	$2\theta_i$	θ_i	$Sin^2(\theta_i)$	$\frac{Sin^2(\theta_i)}{Sin^2(\theta_1)}$	Clear Fraction	$h^2+k^2+l^2$	*hkl*
1	38	19	0.11	1	2	2	110
2	54	27	0.21	1.94	3.88	4	200
3	68	34	0.31	2.95	4.9	6	211
4	80	40	0.41	3.90	7.8	8	220
5	92	46	0.52	4.9	9.8	10	310
6	103	51.5	0.61	5.8	11.6	12	222
7	116	58	0.72	6.8	13.6	14	321

Bravais Lattice: cI

BCC, a=3.397 Å

For following method, i.e., 2theta, theta, sin2theta etc, you give 5 marks
Next look at h2+k2+l2 values and award 4 marks
Correct cyrstal structure: 1 mark
Finally, for correct lattice parameter or approximate value, award 2 marks

For following method, i.e., 2theta, theta, sin2theta etc, you give 5 marks
Next look at h2+k2+l2 values and award 4 marks
Correct cyrstal structure: 1 mark
Finally, for correct lattice parameter or approximate value, award 2 marks

Set No: D

Peaks	$2\theta_i$	θ_i	$Sin^2(\theta_i)$	$\frac{Sin^2(\theta_i)}{Sin^2(\theta_1)}$	Clear Fraction	$h^2+k^2+l^2$	hkl
1	27.1	13.5	0.055	1	3	3	111
2	31.4	15.7	0.073	1.3	3.9	4	200
3	45.3	22.65	0.149	2.7	8.1	8	220
4	53.6	26.785	0.203	3.69	11.07	11	311
5	56.4	28.2	0.223	4.05	12.15	12	222
6	66	33	0.297	5.4	16.2	16	400
7	72.8	36.4	0.353	6.4	19.2	19	331
8	75.4	37.7	0.374	6.8	20.4	20	420
9	83.9	41.95	0.447	8.12	24.36	24	422
10	90.3	45.15	0.503	9.15	27.45	27	511,333

Bravais Lattice: cF

FCC structure, a=5.643 Å

Q3: Mr. Prasad will demonstrate an XRD experiment in today's lab. Please observe and complete the following questions: (5 marks)

(i) What is the target and its characteristic radiation in the machine?

Cu Target, K_alpha (Be lenient in this question, award 1 mark to everyone if they write something)

(ii) What is the scan rate for the samples used?

(Be lenient) Award 1 mark if they write some degree/sec or degree/min value

(iii) Draw the XRD pattern schematic observed (Intensity vs 2θ) from the experiment.

(Be lenient) Award 2 marks if they draw some intensity vs 2\theta

(iv) Draw a schematic of the essential features of an XRD highlighting X-rays from the tube on a crystal and detector measuring the intensity of diffracted X-rays. Highlight the incident and diffracted rays.

(Be lenient) Award 1 mark if they draw some schematic like this