

Laboratory Module 2

INDEXING DIFFRACTION PATTERNS FROM NON-CUBIC MATERIALS

LEARNING OBJECTIVES

Upon completion of this module you will be able to index an X-ray diffraction pattern, identify the Bravais lattice, and calculate the lattice parameters for materials having non-cubic structures.

BACKGROUND

As noted in the previous module, we need to know about crystal structures because structure, to a large extent, determines properties. X-ray diffraction (XRD) is one of a number of experimental tools that are used to identify the structures of crystalline solids. The XRD patterns, the product of an XRD experiment, are somewhat like fingerprints in that they are unique to the material that is being examined. The information in an XRD pattern is a direct result of two things:

- (1) The size and shape of the unit cells which determine the relative positions of the diffraction peaks;
- (2) Atomic positions within the unit cell which determine the relative intensities of the diffraction peaks (remember the structure factor?).

Taking these things into account, we can calculate the size and shape of a unit cell from the positions of the XRD peaks and we can determine the positions of the atoms in the unit cell from the intensities of the diffraction peaks.

Many materials have crystal structures that are not cubic. In other words, they are based on non-cubic Bravais lattices (*e.g.*, hexagonal, tetragonal, orthorhombic, etc...). In fact, many intermetallics, semiconductors, ceramics, and minerals of interest have very complicated structures. Thus, one now needs only to index XRD patterns from non-cubic systems.

How to we correctly index patterns from non-cubic systems? Follow below.

PROCEDURE FOR INDEXING NON-CUBIC XRD PATTERNS

The procedures are standard and will work for any crystal. The equations will differ slightly from each other due to differences in crystal size and shape (*i.e.*, crystal structure). As was the case for cubic crystals, there are two methods of analysis that involve calculations. You will do both. The first is called the “mathematical method.” The second is called the “analytical method.” Both the mathematical and analytical methods require some knowledge of the crystal structure that you are dealing with and the resulting lattice parameter ratios (*e.g.*, c/a , b/a , etc...).

First, consider the plane spacing equations for the crystal structures of interest. Some examples are shown below:

Hexagonal	$\frac{1}{d^2} = \frac{4}{3} \left(\frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2}$
Tetragonal	$\frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2}$
Orthorhombic	$\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}$
Etc.	

You should recall Bragg's law ($\lambda = 2d \sin \theta$), which can be re-written either as:

$$\lambda^2 = 4d^2 \sin^2 \theta$$

or

$$\sin^2 \theta = \frac{\lambda^2}{4d^2}$$

Combining Bragg's law with the plane spacing equations yields the relationship:

Hexagonal	$\frac{1}{d^2} = \frac{4}{3} \left(\frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2} = \frac{4 \sin^2 \theta}{\lambda^2}$
Tetragonal	$\frac{1}{d^2} = \frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2} = \frac{4 \sin^2 \theta}{\lambda^2}$
Orthorhombic	$\frac{1}{d^2} = \frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} = \frac{4 \sin^2 \theta}{\lambda^2}$
Etc...	

which can be rearranged in terms of $\sin^2 \theta$ to:

Hexagonal	$\sin^2 \theta = \left(\frac{\lambda^2}{4} \right) = \left[\frac{4}{3} \left(\frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2} \right]$
Tetragonal	$\sin^2 \theta = \left(\frac{\lambda^2}{4} \right) \left(\frac{h^2 + k^2}{a^2} + \frac{l^2}{c^2} \right)$
Orthorhombic	$\sin^2 \theta = \left(\frac{\lambda^2}{4} \right) \left(\frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2} \right)$

You should note that as unlike cubic systems where $\left(\frac{\lambda^2}{4a^2}\right)$ is constant, your results for non-cubic systems will depend upon ratios of lattice parameters (*i.e.*, c/a , b/a , etc.) and your interaxial angles (*i.e.*, α , β , γ). This is due to the non-equivalence of indices in these systems (*e.g.*, tetragonal – $001 \neq 100$; orthorhombic – $001 \neq 010 \neq 100$; etc...). Since we don't know what it is to start with, we must determine it iteratively. This is done by guessing an initial c/a , indexing using the guessed ratio, calculating lattice parameters, and determining the actual c/a ratio. Then, this value is substituted back in and solved. Using this iterative approach, you should converge towards little or no difference between the experimental and guessed c/a ratios. I know this seems confusing but let's go through some examples below.

Mathematical Method

Let's concentrate on hexagonal systems for the time being. I may ask you to derive relationships for tetragonal and orthorhombic systems in a homework assignment. As noted previously, the mathematical method requires knowledge of the c/a ratio.

Recall the following equation:

$$\sin^2 \theta = \left(\frac{\lambda^2}{4a^2}\right) \left[\frac{4}{3}(h^2 + hk + k^2) + \frac{l^2}{(c/a)^2} \right]$$

Note that the lattice parameter a and the ratio of lattice parameters c/a are constant for a given diffraction pattern. Thus, $\left(\frac{\lambda^2}{4a^2}\right)$ is constant for any pattern. The pattern can now be indexed in by considering the terms in brackets, which are variable:

$$\frac{4}{3}(h^2 + hk + k^2) \tag{1.1}$$

$$\frac{l^2}{(c/a)^2} \tag{1.2}$$

Let's start with term 1 (eq. 1.1). This term only depends on the indices h and k . Thus its value can be calculated for different values of h and k . This is done below for various hk values.

Term 1 calculated for various values of hk

		k			
		0	1	2	3
h	0	0.000	1.333	5.333	12.000
	1	1.333	4.000	9.333	17.333
	2	5.333	9.333	16.000	25.333
	3	12.000	17.333	25.333	36.000

The second term (eq. 1.2) can be determined by substituting in the known c/a ratio. This is illustrated for zinc ($c/a = 1.8563$) in the table below.

Term 2 calculated for zinc ($c/a = 1.8563$)

l	l^2	$l^2l(c/a)^2$
0	0	0.000
1	1	0.290
2	4	1.161
3	9	2.612
4	16	4.643
5	25	7.255
6	36	10.447

The next step is to add the values for the two terms that are permitted by the structure factor (*i.e.*, the values corresponding to the allowed hkl values) and to rank them in increasing order. The structure factor calculation for hexagonal systems yields the following rules:

1. When $h + 2k = 3N$ (where N is an integer), there is no peak.
2. When l is odd, there is no peak.

Both criteria must be met!

Indices (hkl)	l	$h + 2k$	Peak
301	Odd	3	NO
103	Odd	$1 \neq 3N$	YES
Etc...			

Compare with values in appendix 9 in Cullity.

Several values for the bracketed quantity are calculated below minus the peaks forbidden by the structure factor.

<i>h</i>	<i>k</i>	<i>l</i>	<i>sum</i>
0	0	2	1.1608
1	0	0	1.3333
1	0	1	1.6235
1	0	2	2.4942
1	0	3	3.9452
1	1	0	4.0000
0	0	4	4.6433
1	1	2	5.1608
2	0	0	5.3333
2	0	1	5.6235
1	0	4	5.9766
2	0	2	6.4942

The values in this table have been calculated for specific (*hkl*) planes. We can assign specific *hkl* values for each of the peaks in a hexagonal unknown by noting that the sequence of peaks will be the same as indicated in the table.

Lattice parameters can be determined in two ways:

We can calculate *a* by looking for peaks where *l* = 0 (*i.e.*, *hk0* peaks). If you substitute *l* = 0 into:

$$\sin^2 \theta = \left(\frac{\lambda^2}{4a^2} \right) \left[\frac{4}{3} (h^2 + hk + k^2) + \frac{l^2}{(c/a)^2} \right]$$

you will get,

$$\sin^2 \theta = \left(\frac{\lambda^2}{4a^2} \right) \left[\frac{4}{3} (h^2 + hk + k^2) \right]$$

OR

$$a = \frac{\lambda}{\sqrt{3} \sin \theta} \sqrt{h^2 + hk + k^2}$$

You can now perform this calculation for every *hk0* peak, which will yield values for *a*. Similarly, values for *c* can be determined by looking for *00l* type peaks. In these instances, *h* = *k* = 0. Thus,

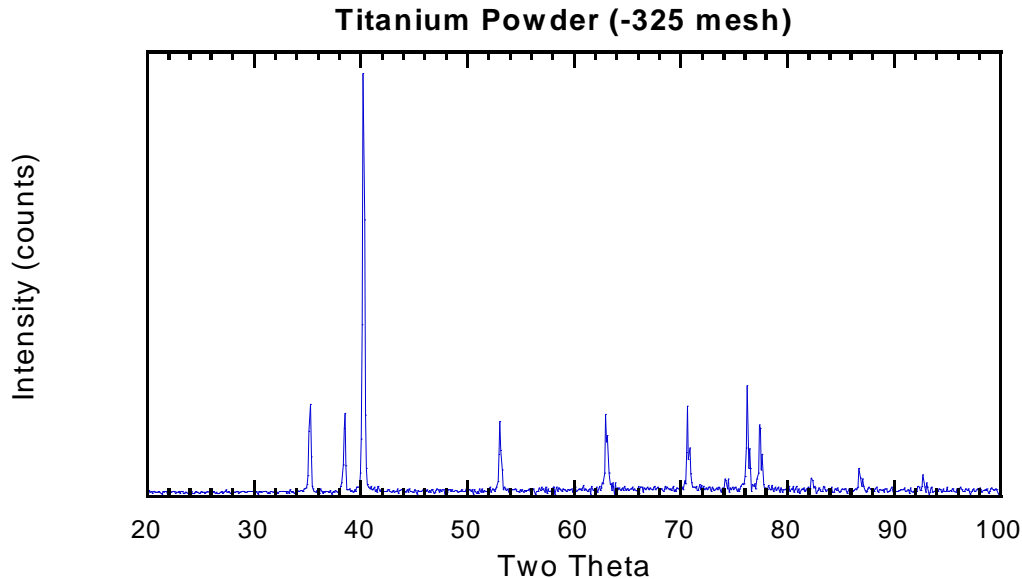
$$\sin^2 \theta = \left(\frac{\lambda^2}{4a^2} \right) \left[\frac{4}{3} (h^2 + hk + k^2) + \frac{l^2}{(c/a)^2} \right]$$

becomes

$$c = \frac{\lambda}{2 \sin \theta} l$$

Worked Example

Consider the following XRD pattern for Titanium, which was collected using $\text{CuK}\alpha$ radiation.



Index this pattern and determine the lattice parameters.

Steps:

(1) Identify the peaks.

(2) Determine values of $\frac{4}{3}(h^2 + hk + k^2)$ for reflections allowed by the structure factor.

(3) Determine values of $\frac{l^2}{(c/a)^2}$ for the allowed reflections and the known c/a ratio

(4) Add the solutions from parts (2) and (3) together and re-arrange them in increasing order.

(5) Use this order to assign indices to the peaks in your diffraction pattern.

(6) Look for $hk0$ type reflections and calculate a for these reflections.

(7) Look for $00l$ type reflections. Calculate c for these reflections.

Here we go!

(1) Identify the peaks.

Peak	l/lo	sin ² θ	d
35.275	21	0.0918	2.542
38.545	18	0.1089	2.334
40.320	100	0.1188	2.235
53.115	16	0.1999	1.723
63.095	11	0.2737	1.472
70.765	9	0.3353	1.330
74.250	10	0.3643	1.276
76.365	8	0.3821	1.246
77.500	14	0.3918	1.231
82.360	2	0.4335	1.170
86.940	2	0.4733	1.120
92.900	10	0.5253	1.063

(2) Determine values of $\frac{4}{3}(h^2 + hk + k^2)$ for reflections allowed by the structure factor.

		k			
		0	1	2	3
h	0	0.000	1.333	5.333	12.000
	1	1.333	4.000	9.333	17.333
	2	5.333	9.333	16.000	25.333
	3	12.000	17.333	25.333	36.000

(3) Determine values of $\frac{l^2}{(c/a)^2}$ for the allowed reflections and the known *c/a* ratio.

Titanium: *c/a* = 1.5871

l	l²	l²(c/a)²
0	0	0.000
1	1	0.397
2	4	1.588
3	9	3.573
4	16	6.352
5	25	9.925
6	36	14.292

(4) Add the solutions from parts (2) and (3) together and re-arrange them in increasing order.

<i>hkl</i>	Pt.1+Pt.2		<i>hkl</i>	Pt.1+Pt.2
002	1.588		100	1.333
100	1.333		002	1.588
101	1.730		101	1.730
102	2.921		102	2.921
103	4.906		110	4.000
110	4.000	→	103	4.906
004	6.352		200	5.333
112	5.588		112	5.588
200	5.333		201	5.730
201	5.730		004	6.352
104	7.685		202	6.921
202	6.921		104	7.685
203	8.906		203	8.906
105	11.258		210	9.333
114	10.352		211	9.730
210	9.333		114	10.352
211	9.730		212	10.921
204	11.685		105	11.258
006	14.292		204	11.685
212	10.921		300	12.000
106	15.625		213	12.906
213	12.906		302	13.588
300	12.000		006	14.292
205	15.258		205	15.258
302	13.588		106	15.625

(5) Use this order to assign indices to the peaks in your diffraction pattern.

Peak	<i>l</i> / <i>o</i>	$\sin^2\theta$	<i>d</i> (nm)	<i>hkl</i>	<i>a</i>	<i>c</i>	h^2+hk+k^2	ρ^2
35.275	21	0.091805	2.5423	100				
38.545	18	0.108941	2.3338	002				
40.320	100	0.118779	2.2351	101				
53.115	16	0.199895	1.7229	102				
63.095	11	0.273744	1.4723	110				
70.765	9	0.335278	1.3303	103				
74.250	10	0.36428	1.2763	200				
76.365	8	0.382132	1.2461	112				
77.500	14	0.39178	1.2307	201				
82.360	2	0.433526	1.1699	004				
86.940	2	0.473309	1.1197	202				
92.900	10	0.525296	1.0628	104				

AVG

(6) Look for $hk0$ type reflections and calculate a for these reflections.

Peak	l/lo	$\sin^2\theta$	d (nm)	hkl	a	c	h^2+hk+k^2	f^2
35.275	21	0.091805	2.5423	100	2.936		1	
38.545	18	0.108941	2.3338	002				
40.320	100	0.118779	2.2351	101				
53.115	16	0.199895	1.7229	102				
63.095	11	0.273744	1.4723	110	2.945		3	
70.765	9	0.335278	1.3303	103				
74.250	10	0.36428	1.2763	200	2.947		4	
76.365	8	0.382132	1.2461	112				
77.500	14	0.39178	1.2307	201				
82.360	2	0.433526	1.1699	004				
86.940	2	0.473309	1.1197	202				
92.900	10	0.525296	1.0628	104				
AVG					2.943		c/a:	

(7) Look for $00l$ type reflections. Calculate c for these reflections.

Peak	l/lo	$\sin^2\theta$	d (nm)	hkl	a	c	h^2+hk+k^2	f^2
35.275	21	0.091805	2.5423	100	2.936		1	
38.545	18	0.108941	2.3338	002		4.668		4
40.320	100	0.118779	2.2351	101				
53.115	16	0.199895	1.7229	102				
63.095	11	0.273744	1.4723	110	2.945		3	
70.765	9	0.335278	1.3303	103				
74.250	10	0.36428	1.2763	200	2.947		4	
76.365	8	0.382132	1.2461	112				
77.500	14	0.39178	1.2307	201				
82.360	2	0.433526	1.1699	004		4.680		16
86.940	2	0.473309	1.1197	202				
92.900	10	0.525296	1.0628	104				
AVG					2.943	4.674	c/a:	1.588

Pretty good correlation with ICDD value. Actual c/a for Titanium is 1.5871

This method requires that you know the c/a ratio for the specimen being evaluated. However if you don't know what it is, you should guess and go through the process. Then, once you calculate lattice parameters, you can calculate the c/a ratio. If you guessed correctly, the experimental values should match those that you guessed. If it does not, use the calculated value as your guess, and go through the process again. You should begin to converge towards the actual value.

The mathematical method, though effective for most powder XRD data, can yield the wrong results if XRD peaks are missing from your XRD pattern. In other words, missing peaks can cause you to assign the wrong hkl values to a peak. Other methods should be available.

Analytical Method

To accurately apply this technique, one must first consider our altered plane spacing equation:

$$\sin^2 \theta = \left(\frac{\lambda^2}{4a^2} \right) \left[\frac{4}{3} (h^2 + hk + k^2) + \frac{l^2}{(c/a)^2} \right]$$

Since a and c/a are constants for any given pattern, we can re-arrange this equation to:

$$\sin^2 \theta = A(h^2 + hk + k^2) + Cl^2$$

where $A = \frac{\lambda^2}{3a^2}$ and $C = \frac{\lambda^2}{4c^2}$. Since h , k , and l are always integers, the term in parentheses, $h^2 + hk + k^2$ can only have values like 0, 1, 3, 4, 7, 9, 12... and l^2 can only have values like 0, 1, 4, 9,....

We need to calculate $\sin^2 \theta$ for each peak, divide each $\sin^2 \theta$ value by the integers 3, 4, 7, 9... and look for the common quotient (*i.e.*, the $\sin^2 \theta / n$ value that is equal to one of the observed $\sin^2 \theta$ values). The $\sin^2 \theta$ values representing this common quotient refer to $hk0$ type peaks. Thus this common quotient can be tentatively assigned as A .

We can now re-arrange terms in our modified equation to obtain C . This is done as follows:

$$\begin{aligned} \sin^2 \theta &= A(h^2 + hk + k^2) + Cl^2 \\ &\Downarrow \\ Cl^2 &= \sin^2 \theta - A(h^2 + hk + k^2) \end{aligned}$$

We get the value of C by subtracting from each $\sin^2 \theta$ the values of $n \cdot A$ (*i.e.*, $A, 3A, 4A, 7A, \dots$) where A is the common quotient that we identified above. Next, we need to look for the remainders that are in the ratio of 1, 4, 9, 16..., which will be peaks of the $00l$ type. We can determine C from these peaks. The remaining peaks are neither $hk0$ -type nor $00l$ type. Instead they are hkl -type. They can be indexed from a combination of A and C values. Let's do an example.

Worked Example

Steps:

- (1) Identify the peaks and calculate $\sin^2 \theta$ for each peak.
- (2) Divide each $\sin^2 \theta$ value by the integers 3, 4, 7, 9,....
- (3) Look for the common quotient.
- (4) Let the lowest common quotient represent A .

- (5) Assign $hk0$ type indices to peaks.
- (6) Calculate $\sin^2 \theta - nA$ where $n = 1, 3, 4, 7, \dots$
- (7) Look for the lowest common quotient. From this we can identify $00l$ type peaks. Recall, that 001 is not allowed for hexagonal systems. The first $00l$ type peak will be 002 . We can calculate C from:

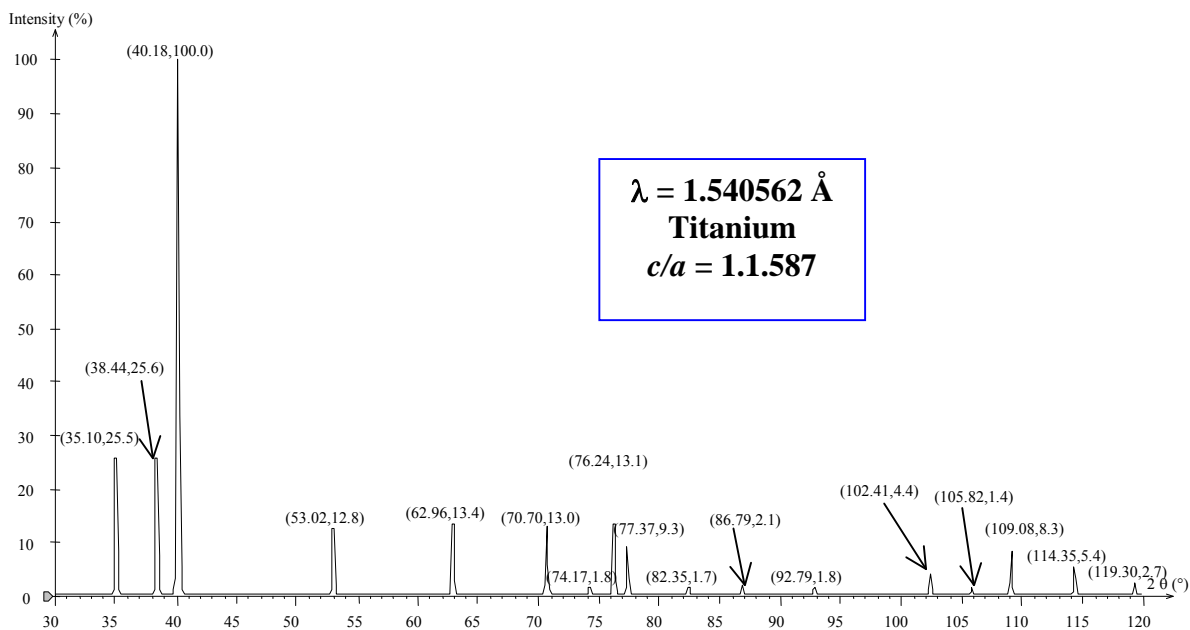
$$C \cdot l^2 = \sin^2 \theta - A \cdot (h^2 + hk + k^2)$$

- (8) Look for values of $\sin^2 \theta$ that increase by factors of 4, 9... (this is because $l = 1, 2, 3, \dots$ and $l^2 = 1, 4, 9, \dots$). Peaks exhibiting these characteristics are $00l$ type peaks, which can be assigned the indices $004, 009, \dots$). Also note that the values of $\sin^2 \theta$ will be some integral number times the value observed in (7) which indicates the indices of the peak
- (9) Peaks that are neither $hk0$ nor $00l$ can be identified using combinations of our calculated A and C values.
- (10) Calculate the lattice parameters from the values of A and C .

Confused yet? You could be. I was the first time I learned these things. Let me show you an example that should make all things clear.

Here we go!

Consider the diffraction pattern for Titanium as shown below. This one is a little different than the specimen that we analyzed above.



Steps to success:

1. Calculate $\sin^2\theta$ for each peak
2. Divide each $\sin^2\theta$ value by integers 3, 4, 7...
(from h^2+hk+k^2 allowed by the structure factor)
3. Look for lowest common quotient.
4. Let lowest common quotient = A.
5. Peaks with lowest common quotient are $hk0$ type peaks.
Assign allowed $hk0$ indices to peaks.

For this part of the problem

$$\frac{\sin^2 \theta}{n} = \frac{\sin^2 \theta}{(h^2 + hk + k^2)}$$

Peak	l/lo	$\sin^2\theta$	$(\sin^2\theta)/3$	$(\sin^2\theta)/4$	$(\sin^2\theta)/7$	$(\sin^2\theta)/9$	$(\sin^2\theta)/12$	hkl	$(\sin^2\theta)/A$
35.100	25.5	0.0909	0.0303	0.0227	0.0130	0.0101	0.0076	100	1.0
38.390	25.6	0.1081	0.0360	0.0270	0.0154	0.0120	0.0090		1.2
40.170	100	0.1179	0.0393	0.0295	0.0168	0.0131	0.0098		1.3
53.000	12.8	0.1991	0.0664	0.0498	0.0284	0.0221	0.0166		2.2
62.940	13.4	0.2725	0.0908	0.0681	0.0389	0.0303	0.0227	110	3.0
70.650	13	0.3343	0.1114	0.0836	0.0478	0.0371	0.0279		3.7
74.170	1.8	0.3636	0.1212	0.0909	0.0519	0.0404	0.0303	200	4.0
76.210	13.1	0.3808	0.1269	0.0952	0.0544	0.0423	0.0317		4.2
77.350	9.3	0.3905	0.1302	0.0976	0.0558	0.0434	0.0325		4.3
82.200	1.7	0.4321	0.1440	0.1080	0.0617	0.0480	0.0360		4.8
86.740	2.1	0.4716	0.1572	0.1179	0.0674	0.0524	0.0393		5.2
92.680	1.8	0.5234	0.1745	0.1308	0.0748	0.0582	0.0436		5.8
102.350	4.4	0.6069	0.2023	0.1517	0.0867	0.0674	0.0506		6.7
105.600	1.4	0.6345	0.2115	0.1586	0.0906	0.0705	0.0529	210	7.0
109.050	8.3	0.6632	0.2211	0.1658	0.0947	0.0737	0.0553		7.3
114.220	5.4	0.7051	0.2350	0.1763	0.1007	0.0783	0.0588		7.8
119.280	2.7	0.7445	0.2482	0.1861	0.1064	0.0827	0.0620		8.2

A = **0.0908**

Indices correspond to:
 $h^2+hk+k^2 = 1, 3, 4, 7, \dots$
 or
 $hk = 10, 11, 20, 21$

Crystal structure determination for non-cubic crystals

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6. Subtract from each $\sin^2\theta$ value $3A, 4A, 7A, \dots$
(from h^2+hk+k^2 allowed by the structure factor);
7. Look for lowest common quotient (LCQ). From this you can identify $00l$ -type peaks. The first allowed peak for hexagonal systems is 002 . Determine C from the equation:
 $C \times l^2 = \sin^2\theta - (h^2+hk+k^2)A$
 Since $h=0$ and $k=0$, then:
 $C = LCQ/l^2 = \sin^2\theta/l^2$;
8. Look for values of remainders that increase by factors of 1, 4, 9, 16...
(because $l = 1, 2, 3, 4, \dots, l^2 = 1, 4, 9, 16, \dots$). The peaks exhibiting these characteristics are allowed $00l$ -type peaks (e.g., $002, 004, \dots$).

- We identify the 4th peak as 102 because we observe the LCQ for $\sin^2\theta-1A$. Recall that the 1 comes from the quadratic form of Miller indices (i.e., $h^2+hk+k^2=1$).
- We identify the 8th peak as 112 because we observe the LCQ for $\sin^2\theta-3A$. Recall that the 1 comes from the quadratic form of Miller indices (i.e., $h^2+hk+k^2=3$).
- We identify the 11th peak as ...
- etc...

λ	h^2+hk+k^2	0	1	3	4	7	A = 0.0908	h	k	l	C = LCQ/l ²	l ² = LCQ/C
1.54062												
35.100	25.5	0.0909						1	0	0		
38.390	25.6	0.1081	0.0173					1.0	0	2	0.0270	4.0
40.170	100	0.1179	0.0271					1.1				
53.000	12.8	0.1991	0.1083					1.8	1	0	0.0271	
62.940	13.4	0.2725	0.1817	0.0001				1	1	0		
70.650	13	0.3343	0.2435	0.0618				3.1				
74.170	1.8	0.3636	0.2728	0.0911	0.0003			2	0	0		
76.210	13.1	0.3808	0.2900	0.1083	0.0175			3.5	1	1	0.0271	
77.350	9.3	0.3905	0.2997	0.1180	0.0272			3.6				
82.200	1.7	0.4321	0.3413	0.1597	0.0688			4.0	0	0	0.0270	16
86.740	2.1	0.4716	0.3807	0.1991	0.1083			4.4	2	0	0.0271	
92.680	1.8	0.5234	0.4326	0.2509	0.1601			4.8				
102.350	4.4	0.6069	0.5161	0.3345	0.2436			5.6				
105.600	1.4	0.6345	0.5436	0.3620	0.2711			2	1	0		
109.050	8.3	0.6632	0.5724	0.3907	0.2999	0.0274		6.1				
114.220	5.4	0.7051	0.6143	0.4326	0.3418	0.0693		6.5				
119.280	2.7	0.7445	0.6537	0.4721	0.3812	0.1087		6.9				

LCQ = **0.1083**

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$$\sin^2 \theta = Cl^2 + A(h^2 + hk + k^2)$$

Cycle through allowed values for h , k and l , and compare the $\sin^2\theta$ value to the labeled peaks. They are the hkl peaks.

A		C					
0.0908		0.0270					
Peak	l/λ	$\sin^2\theta$	h	k	l	$\sin^2\theta$ Calculated	$= Cl^2 + A \times (h^2 + hk + k^2)$
35.100	25.5	0.0909	1	0	0	0.0908	
38.390	25.6	0.1081	0	0	2	0.1081	
40.170	100.0	0.1179	1	0	1	0.1179	
53.000	12.8	0.1991	1	0	2	0.1989	
62.940	13.4	0.2725	1	1	0	0.2725	
70.650	13.0	0.3343	1	0	3	0.3341	
74.170	1.8	0.3636	2	0	0	0.3633	
76.210	13.1	0.3808	1	1	2	0.3806	
77.350	9.3	0.3905	2	0	1	0.3903	
82.200	1.7	0.4321	0	0	4	0.4324	
86.740	2.1	0.4716	2	0	2	0.4714	
92.680	1.8	0.5234	1	0	4	0.5232	
102.350	4.4	0.6069	2	0	3	0.6065	
105.600	1.4	0.6345	2	1	0	0.6358	
109.050	8.3	0.6632	2	1	1	0.6628	
114.220	5.4	0.7051	1	1	4	0.7049	
119.280	2.7	0.7445	2	1	2	0.7439	

10. Now that we know A and C , we can calculate lattice parameters.

$$a = \frac{\lambda}{\sqrt{3A}} \quad c = \frac{\lambda}{\sqrt{4C}} \quad \rightarrow \quad \begin{array}{|c|c|c|} \hline a & c & c/a \\ \hline 2.951 & 4.686 & 1.588 \\ \hline \end{array}$$

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LABORATORY INSTRUCTIONS/OBJECTIVES

Each laboratory group will collect an XRD pattern from a cubic crystal powder (assigned by the instructor). Data should be collected over the angular range 15 to 120 degrees two theta with a collection step size of 0.02 and a collection time of 1s/step. The data is to be indexed using both the mathematical and analytical methods. Lattice parameters are to be estimated based upon the experimental data. Written lab reports are to be turned in using the Laboratory Template provided on the course website. The written report should briefly describe the steps/conditions taken to prepare specimens, collect, data, and analyze/index the data. any differences between the collected data and standard results (i.e., ICDD reference cards) should be discussed. The objective of this exercise is to teach you how to properly collect, interpret, and report scientific (i.e., XRD) data.