Diffractometric Debye-Scherrer patterns of powder samples with a cubic powder sample

Objectives:

1-To determine the lattice constant a of an unknown material .

2-Assign the Bragg reflexes to the respective lattice planes. Determine which Bravais lattice type it has .

3-Determine the type of the unknown material.

Theory:

When X-rays of wavelength strikes a mass of lattice planes of a crystal at a glancing angle θ ,the reflected rays will only be subject to constructive interference when Bragg condition is fulfilled .Bragg's condition implies that all of the waves scattered at the atom are in phase and so amplify each other, whereas partial waves that are scattered in directions not fulfilling Bragg's condition are of opposite phase and so extinguishing each other. A more realistic way of looking at this must,however,take the actual phase relationships of all the partial waves scattered by the atom in certain direction into consideration. When there are N atoms in a unit cell, then the total amplitude of the X-rays scattered by the cell is described by the structure factor ***F*** ,which is the sum of the atomic scattering factors ***f*** of the individual N atoms, taking their phases into account, in general the following is valid for ***F*****:**

$$F_{hkl} = \sum_1^N f_n \cdot e^{2\pi i(hu_n + kv_n + lw_n)} \qquad (1)$$

where h, k, l =Miller indices of the reflecting lattice planes and u_n , v_n, w_n are the coordinates of the atoms in fractions of the particular edge lengths of the unit cell.

as F in general a complex numbe,the total scattering intensity is described by $|F_{hkl}|^2$.A cubic simple unit cell contains only one atom with coordinate 000.From equation 1 therefore the structure factor for this lattice type is given by:

$$F = f.e^{2\pi i(0)} = f;\ |F|^2 = f^2 \qquad (2)$$

this means that F^2 is independent of $h, k\ and\ l$ and all Bragg reflexes can occur.

the unit cell of a cubic face-centered lattice has 4 atoms at $000, {}^1/_2\ , {}^1/_2\ ,0\ and\ 0, {}^1/_2\ {}^1/_2$ the unit cell of a cubic body-centerd lattice has in comparison only 2 atoms at 000,and $ {}^1/_2, {}^1/_2\ {}^1/_2$ should the lattice only consist of one sort of atom, then the following conditions are given for the structure factor:

fcc lattice

$|F|^2 = 16\,f^2$ with hkl only even or only odd

$|F|^2 = 0$ with hkl mixed

bcc lattice

$|F|^2 = 4\,f^2$ with($h + k + l$) even

$|F|^2 = 0$ with($h + k + l$) odd

the situation is a little different when a lattice is constructed of different sorts of atoms.

for a cubic crystal system, the spacing d of the individual lattice planes with the indices(hkl) is obtained from the quadratic form:

$\frac{1}{d_{hkl}^2} = \frac{1}{a^2}(h^2 + k^2 + l^2\ )$ (where a is the lattice constant) (3)

from this and Bragg equation $n\lambda=2d\ \sin\theta$ with n=1 the quadratic Bragg equation is obtained:

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

the following so-called strip-matching procedure can be used to index the individual reflexes of cubic crystals. Take logarithms in equation (4):

$$loga = \log\left(\frac{1}{2}\lambda\right) + \log\ (\sqrt{h^2 + k^2 + l^2}\ \text{-log(sin}\theta) \quad (5)$$

Plot the experimentally determined values for log(sinθ) on a strip of paper in addition,plot the 2nd term on the right hand side of equation (5)on a seperate paper,taking all possible index triplets into considerations

$$\log\left(\frac{1}{2}\lambda\right) + \log\ (\sqrt{h^2 + k^2 + l^2}\ \quad (9)$$

Now move the scales against each other until a position is found at which the graduations on the two stripes match up to a great extent.The distance between the zero points of the two stripes now gives the value of log(a).Taking anti-logsgives the lattice constant a of the cubic system. table 1 gives the permissible hkl index triplets for fcc and bcc lattices.

Table 1: permissible hkl index triplets for fcc and bcc lattices for molybdenum

h k l	$h^2+k^2+l^2$	$log\sqrt{h^2+k^2+l^2} + \log(\lambda/2)$
111	3	1.789278051
200	4	1.851747419
112	6	1.939793049
022	8	2.002262417
013	10	2.050717423
113	11	2.071413766
222	12	2.090308046
213	14	2.123781441
004	16	2.152777415
114	18	2.178353676
033	18	2.178353676
313	19	2.190094224
024	20	2.201232421
323	22	2.221928764
224	24	2.240823044

Apparatus:

X-ray apparatus -powder sample 0f unknown material -Pc with X-ray apparatus program.

Procedure:

1-start the software "X-ray apparatus", and clear any existing data.
2-set the high voltage U=35 kV , the emission current I =1 mA ,the measuring time per angular step Δt=10s and the angular step width $\Delta\beta = 0.1^{0}$
3-Press the COUPLED key to activate 2 θ coupling of target and sensor and set the lower limit of the target angle to 7^{0} and the upper limit to 30^{0}.
4-start measurement and data transfer to the PC by pressing the SCAN key.
5--when you have finished measuring save the measurement.
6-in each diffraction diagram use the left mouse button ,mark the "full width "of each peak and write down the centre values in a table as the glancing angle.
7-Press the zero key to return the target and sensor to the current zero point.

Measurements:

Line	θ	Sinθ	-log sinθ
1			
2			
3			
4			
5			

Results:

-use the strip-matching method (fig.1) to find the type of lattice ,lattice constant ,and the unknown material.

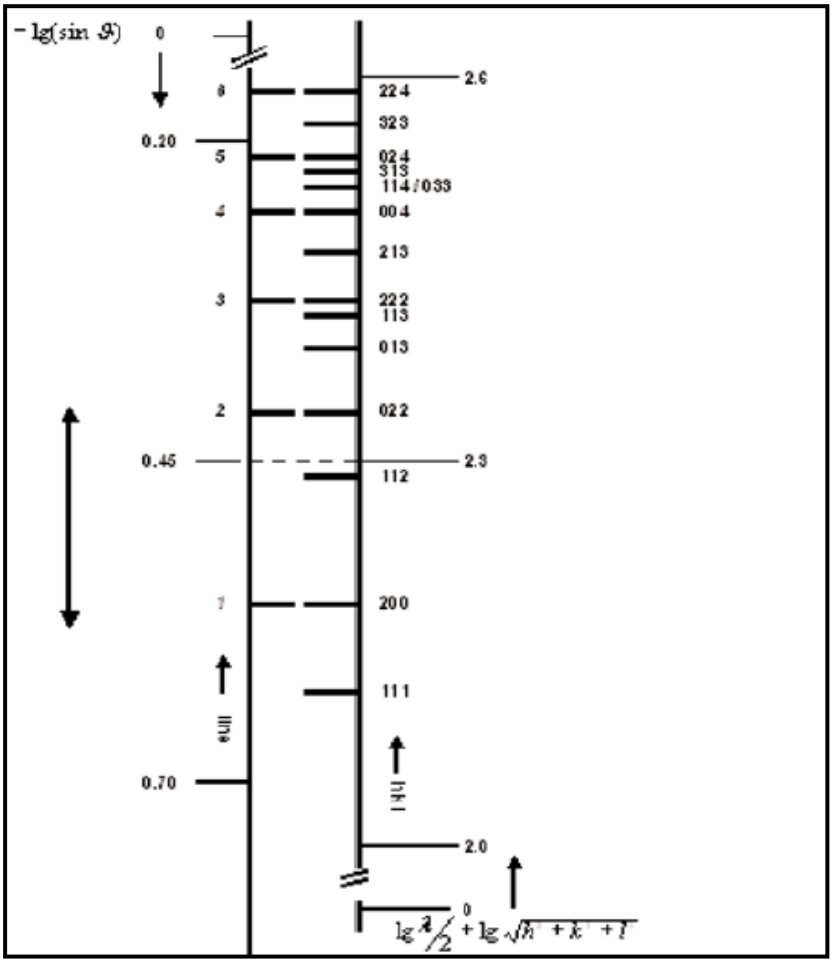

Fig.1 the strip-matching method