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3. MEASUREMENT OF INTER PLANNER SPACING OF SODIUM CHLORIDE CRYSTAL

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Abstract :- A novel diffraction method is used for the measurement of the inter planner spacing of Sodium Chloride with accuracy is presented. This method is mainly based on Bragg's X-Ray Diffraction Method which is carried out by a device called Goniometer (Contact angle type) along with an X-Ray source and a receiver. In this experiment the wave length from a sealed X-ray source is used in a high quality precision Goniometer to obtain the direct measurement of the diffracted angle in a single determination. This method is applicable to a range of material types from a perfect single crystal to polycrystalline samples because it involves a single measurement.

Key Words : Goniometer, Bragg's Diffraction, Unit Cell, FCC Structure

Introduction :

The inter planner spacing plays a vital role in determining the crystal structure[1]. Knowledge of the process of determining inter planner spacing is of crucial importance for the researchers in the field of structure of crystals. Though there are many methods depending upon the nature of the crystal, Goniometer[2] experiment has its own value. In this experiment a sealed X-Ray source called transmitter is fixed on one arm of the goniometer and a receiver which receives the diffracted X-ray and measure the intensity of diffracted beam in terms of current (mA) is fixed on the other arm at an equal distance from the centre of the gonoimeter(contact angle type) The crystal structure of a material or the arrangement of atoms in a crystal structure can be described in terms of its unit cell[3]. The unit cell is a tiny box

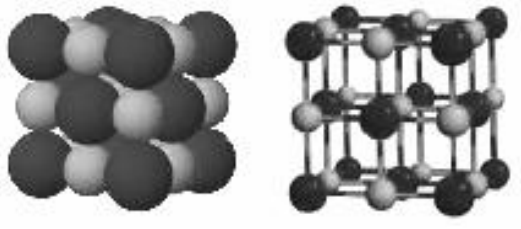

Fig.-1- A model of a sodium chloride crystal, showing how the sodium ions Na^+(small spheres) and chloride ions Cl^-(large spheres) are stacked in the unit cell, whose edge 'a' has the length 0.558nm.

containing one or more motifs[3], a spatial arrangement of atoms. The unit cells stacked in three-dimensional space describe the bulk arrangement of atoms of the crystal. The unit cell is given by its lattice parameters, the length of the cell edges and the angles between them. The crystallographic directions are fictitious lines linking nodes (atoms, ions or molecules) of a crystal. The crystallographic planes[3],[4] are fictitious planes linking nodes. Some directions and planes have a higher density of nodes; these dense planes have an influence on the behavior of the crystal. The smallest distance between two crystallographic planes is known as inter planner spacing.

Crystal Structure of Sodium Chloride:

Sodium Chloride, NaCl, commonly known as rock salt. Sodium Chloride forms isometric crystals[3]. The mineral is typically color less. It commonly occurs with other minerals such as several of the sulphates, halides and borates. Sodium chloride, also known as common salt, table salt, is a chemical compound with the formula NaCl. Sodium chloride is the salt most responsible for the salinity of the ocean. In the structure of NaCl each of the two atom types forms a separate face-centered cubic lattice, with the two lattices interpenetrating so as to form a 3D checkerboard pattern. In the Sodium chloride (NaCl) structure, the space lattice is face centered cubic (FCC). There are two such FCC lattices[3] which are separated by one half of the body diagonal of the unit cube. One lattice is occupied by Na atoms, the other by Cl atoms. One of chlorine ions having its origin at the (0,0,0) point and the other of the Sodium ions having its origin midway along the cube edge at (a/2,0,0) point. Even though there is an attractive force between Na ion and Cl ion they can not coincide due to repulsive force arises at a particular distance between the two ions and it is short range. The fig-1- shows how sodium and chlorine atoms (strictly, Na^+ and Cl^- ions) are stacked to form a crystal of sodium chloride. This pattern, which has cubic symmetry is one of the many possible atomic arrangements exhibited by solids. The model represents the unit cell for sodium chloride. This is the smallest unit from which the crystal may be built up by repetition in three dimensions.

The co-ordination number[3],[4] is defined as the number of nearest atoms to any atom. In case of Sodium Chloride each atom has 6 nearest neighbors of the opposite kind and is bound to these 6 atoms which are arranged at the corners of the surrounding octahedron. So, co-ordination number is 6. The bonding is typically ionic. There are eight Cl ion at the corner of the unit cell and six Cl ions are at the intersecting points of face diagonals. Hence the number of Cl ions per unit cell is 4. There are 12 Na ions at the edges of the unit cell and one more Na ion is at the center of the unit cell. So the number of Na ions per unit cell is 4. Hence the number of Na ions per unit cell is 4. Thus there are four NaCl molecules per unit cell.

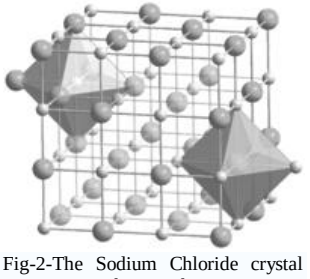

Fig-2-The Sodium Chloride crystal structure. Each atom has six nearest neighbors, with octahedral geometry.

X-Ray Diffraction and Bragg's Law:

Since a beam of X-ray consists of a bundle of separate waves, the waves can interact with one another. Such interaction is termed interference[5]. If all the waves in the bundle are in phase, that is their crests and troughs occur at exactly the same position (the same as being an integer

number of wavelengths out of phase 'nλ' where n = 1, 2, 3, 4, etc.), the waves will interfere with one another and their amplitudes will add together to produce a resultant wave that has a higher amplitude (the sum of all the waves that are in phase.)

If the waves are out of phase, being off by a non-integer number of wavelengths, then destructive interference[6],[2] will occur and the amplitude of the waves will be reduced. In an extreme case, if the waves are out of phase by a multiple of 1/2λ (n/2λ), the resultant wave will have no amplitude and thus be completely destroyed.

The atoms in crystals interact with X-ray waves in such a way as to produce interference. The interaction can be thought of as if the atoms in a crystal structure reflect the waves. But, because a crystal structure consists of an orderly arrangement of atoms, the reflections occur from what appears to be planes of atoms. Let's imagine a beam of X-rays entering a crystal with one of these planes of atoms oriented at an angle θ to the incoming beam of monochromatic[3],[6] X-rays (monochromatic means one color, or in this case one discrete wavelength as produced by the characteristic spectra of the X-ray tube).

Two such X-rays are shown in the fig-3, where the spacing between the atomic planes occurs over the distance 'd'. Ray-1 reflects off of the upper atomic plane at an angle θ equal to its angle of incidence. Similarly, Ray-2 reflects off the lower atomic plane at the same angle θ. While Ray-2 is in the crystal, however, it travels a distance of 2a farther than Ray-1 i,e the path difference between the Ray-1 and Ray-2 is 2a. If this distance 2a is equal to an integral number of wavelengths (nλ) then Rays 1 and 2 will be in phase on their exit from the crystal and constructive interference[6],[2] will occur.

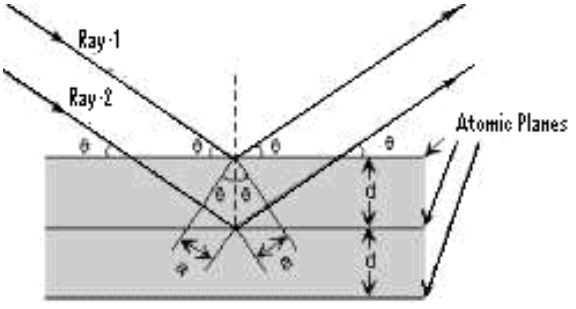

Fig-3- Bragg's Diffraction

If the path difference i,e the distance 2a is not an integral number of wavelengths, then destructive interference will occur and the waves will not be as strong as when they entered the crystal. Thus, the condition for constructive interference to occur is $n\lambda = 2a$ but, from trigonometry, we can write the distance 2a in terms of the spacing 'd' between the atomic planes as,

$$a = d \sin \theta$$

or $2a = 2 d \sin \theta$,

thus, $2d \sin \theta = n\lambda$

This is known as Bragg's Law[2],[5] for X-ray diffraction.

What it says is that if we know the wavelength 'λ' of the X-rays going in to the crystal, and by measuring the angle θ of the diffracted X-rays coming out of the crystal, we can calculate the inter planner spacing (referred to as d-spacing) between the atomic planes by using the formula

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

Again it is important to point out that this diffraction will only occur if the rays are in phase when they emerge, and this will only occur at the appropriate value of n (1, 2, 3, etc.) and θ.

In theory, then we could re-orient the crystal so that another atomic plane is exposed and measure the d-spacing between all atomic planes in the crystal.

Procedure:

Here the object is to plot a graph between intensity vs. grazing angle for two different Bragg's plane.

1. Straighten the goniometer arms such that Transmitter and receiver face each other directly then will be at 0 and 180 degrees.
2. The X-Ray source is fixed on one arm and the receiver is fixed at other arm and at an equal distance from the fulcrum of the goniometer.
3. Rotating table is kept on the goniometer and is rotated such that the angle indicator reads 0 degrees. Here grazing angle[1] is noted not the angle of incidence. Grazing angle is the angle between the plane surface and the incident ray. It is the complement of incident angle.
4. Now NaCl crystal is kept on the rotating table in the center of the Goniometer such that the Bragg's plane (1,0,0) is parallel to both the analyzer and source.
5. Transmitter AC adapter is connected and receiver is on. Multiplier and fine-tuning knobs are adjusted on the receiver so that one can read 0.5 on the scale. One have to so aware that nothing (arms, head, books etc) was close to the apparatus while making the reading to minimize the reflections which may cause interference and give false measurements. Data is entered in the table in the hand in sheet starting off with 0 degrees and 0.5 as my first data pair.
6. Rotating table was turned and hence the crystal by one degree clockwise and the movable arm of goniometer by two degrees clockwise. Here one should maintain the incident angle the same as the reflected angle, a condition necessary for Bragg's diffraction[2]. Readings are noted. The procedure is repeated each time rotating table one degree (two degrees for movable arm) to take new reading.

The graph is drawn by taking 2θ along X-axis and intensity(mA) in Y-axis the nature of the graph is like the fig-4- and the peaks in the graph is noted. Each peak corresponds to maxima in the $2d \sin\theta = n\lambda$ equation. The higher order maxim has greater angles. From the data the inter planar spacing between consecutive Bragg's planes(1,0,0) is calculated. Repeating the same procedure for other planes the inter planner distance for respective plane can be determined.

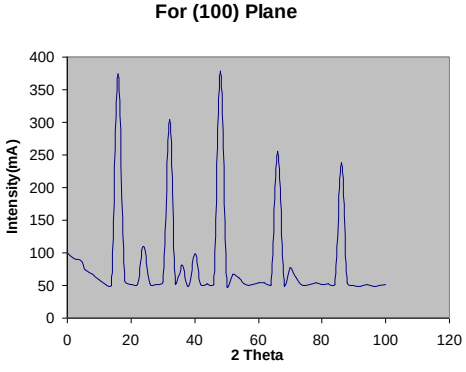

Fig.- 4 - Graph is drawn by taking 2θ along X- axis and corresponding current in mA along Y-axis for (100) plane

Result :-

The above experiment was conducted by taking the X-ray of wave length 0.1542 nm.
From the graph for n = 1, $2\theta = 16^0 \implies \theta = 8^0$

So, $d = \frac{n\lambda}{2\sin\theta} = \frac{0.1542}{2\sin 8^0} = \frac{0.1542}{2 \text{ x } 0.13973} = 0.552\text{nm}$

From theory the lattice parameter for NaCl , a = 0.558nm, the Miller Indices are h = 1, k = 0, *l* = 0 for (1,0,0)plane and inter planner spacing 'd' can be calculated by using the formula,

$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}} = \frac{0.558}{\sqrt{1^2 + 0^2 + 0^2}} = 0.558\text{nm}$ Which satisfy the experimental value.

Comparison Table :-

In the following table experimental value of 'θ' for different planes of Sodium Chloride is given and the corresponding 'd' is calculated(rounded to three decimal places) and compared with theoretical result.

	Experimental Values			**Theoretical Value**
Plane (h,k,l)	2θ in degrees	θ in degrees	d = λ /(2sinθ) in nm	$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$ nm
(2,0,0)	32	16	0.279	0.279
(2,2,0)	46	23	0.197	0.197
(2,2,2)	56	28	0.164	0.161
(2,1,1)	40	20	0.225	0.227
(3,1,0)	52	26	0.176	0.176

Table - 1
(a = 0.558nm, λ = 0.1542nm, n = 1)

Conclusion:-

This is a very simple Method and can be easily assembled in research laboratory with small investment to measure the inter planner spacing between the crystal planes, hence to study the crystal structure

Acknowledgment

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