Crystal Structure Analysis

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Abstract

X-Ray powder diffraction measurement, is a method for structural analysis of an unknown crystal, by investigation the X-ray diffraction angles and intensity from randomly oriented powder crystallites irradiated by a monochromated X-ray beam. In this diffraction process, electrons in the component atoms of crystallites are forced to vibrate by incident X-ray beam, and make sharp coherent scatters of the X-ray. The diffraction pattern from crystalline specimen is specific for the crystal form of the specimen. Therefore, the X-ray powder diffraction method can be used for qualitative and quantitative evaluation of crystal forms.

In powder or polycrystalline diffraction, it is important to have a sample with a smooth plane surface. The ideal sample is homogeneous and the crystallites are randomly distributed. The sample is pressed into a sample holder so that we have a smooth flat surface.

In this project the crystal structure of some different binary system have been successfully determined from their pattern diffraction spectrums take place by the monochromatic X-ray ($\lambda=0.193609 \text{ nm}$). After finding, the miler indexes ($hkl$) of the plane that caused diffraction from all peaks in the spectrum using the idea of geometric factor, the lattice constant can be founded easily using the Bragg’s law. The analysis of the spectrums shows that the (BaO, NaO$_2$, MgS) have Face Centered Cubic structures, while the samples of (ZnZr, AgCd, CuSc) have Body Centered Cubic crystal structures; finally the samples of (LaTe, ErTe) have Simple Cubic structures.
Chapter One
Crystal Structure

1.1 Introduction:

The study of solid state physics began in the early years of twentieth century following the discovery of x-ray diffraction by crystals and the publication of a series of simple calculations and successful predictions of the properties of crystals. Solids can be classified into three categories according to their structure; amorphous, crystal, and polycrystal. The first type an amorphous solid is a solid in which there is no long-range order of the positions of the atoms. (Solids in which there is long-range atomic order are called crystalline solids or amorphous). Most classes of solid materials can be found or prepared in an amorphous form. For instance, common window glass is an amorphous ceramic, many polymers (such as polystyrene) are amorphous, and even foods such as cotton candy are amorphous solids.

In materials science, a crystal is a solid substance in which the atoms, molecules, or ions are arranged in an orderly repeating pattern extending in all three spatial dimensions; while the polycrystalline materials are solids that are composed of many crystallites of varying size and orientation. The variation in direction can be random (called random texture) or directed, possibly due to growth and processing conditions. Fiber texture is an example of the latter.

Almost all common metals, and many ceramics are polycrystalline. The crystallites are often referred to as grains; however, powder grains are a different context. Powder grains can themselves be composed of smaller polycrystalline grains.

Polycrystalline is the structure of a solid material that, when cooled, form crystallite grains at different points within it. Where these crystallite grains meet is known as grain boundaries.
1.2 Crystal structure:

In mineralogy and crystallography, a crystal structure is a unique arrangement of atoms in a crystal. A crystal structure is composed of a motif, a set of atoms arranged in a particular way, and a lattice. Motifs are located upon the points of a lattice, which is an array of points repeating periodically in three dimensions. The points can be thought of as forming identical tiny boxes, called unit cells, that fill the space of the lattice. The lengths of the edges of a unit cell and the angles between them are called the lattice parameters. The symmetry properties of the crystal are embodied in its space group. A crystal's structure and symmetry play a role in determining many of its properties, such as cleavage, electronic band structure, and optical properties.

1.3 The characterization of crystal structures:-

Knowing that the translational operations repeat the content of the unit cell at infinity, we have only to specify the content of a single unit cell. The following information should be specify

- The chemical formula and number of formula units contained in the cell
- The dimensions of the cell
- The space group (and crystal system which can be deduced from the space group)
- The cell content

The number of parameters to specify the dimensions of the cell vary with the crystal system. Only one parameter is sufficient for a cubic cell (the cell length) whereas six parameters are necessary for a triclinic cell (a, b, c, α, β and γ). For the other systems, the numbers of cell parameters vary between these extremes.

Any atom placed in the cell will be reproduced a number of times according to the space group operations. Therefore, only symmetry
independent atoms have to be specified. The coordinates of each symmetry
independent atoms are given in fraction of the cell length. For each atom, the
number of free variables depends on the Wyckoff positions of the space group
and may vary from three (general position) to zero (special position).

1.4 Unit cell:
The crystal structure of a material or the arrangement of atoms in a
crystal structure that can be described in terms of its unit cell. The unit cell is
a tiny box containing one or more motifs, a spatial arrangement of atoms. The
units cells stacked in three-dimensional space describes the bulk arrangement
of atoms of the crystal structure has a three dimensional shape. The unit cell is
given by its lattice parameters, the length of the cell edges and the angles
between them, while the positions of the atoms inside the unit cell are
described by the set of atomic positions \((x_i, y_i, z_i)\) measured from a lattice
point.

Although there are an infinite number of ways to specify a unit cell, for
each crystal structure there is a conventional unit cell, which is chosen to
display the full symmetry of the crystal [see Figure (1.1)]. However, the
conventional unit cell is not always the smallest possible choice. A primitive
unit cell of a particular crystal structure is the smallest possible volume one
can construct with the arrangement of atoms in the crystal such that, when
stacked, completely fills the space. This primitive unit cell will not always
display all the symmetries inherent in the crystal. A Wigner-Seitz cell is a
particular kind of primitive cell which has the same symmetry as the lattice.

In a unit cell each atom has an identical environment when stacked in 3
dimensional spaces. In a primitive cell, each atom may not have the same
environment. Unit cell definition using parallelepiped with lengths a, b, c and
angles between the sides given by \(\alpha, \beta, \gamma\).
1.5 Classification of crystals by symmetry:

The defining property of a crystal is its inherent symmetry, by which we mean that under certain operations the crystal remains unchanged. For example, rotating the crystal 180 degrees about a certain axis may result in an atomic configuration which is identical to the original configuration. The crystal is then said to have a twofold rotational symmetry about this axis. In addition to rotational symmetries like this, a crystal may have symmetries in the form of mirror planes and translational symmetries, and also the so-called compound symmetries which are a combination of translation and rotation/mirror symmetries. A full classification of a crystal is achieved when all of these inherent symmetries of the crystal are identified.

1.6 Crystal system:

The crystal systems are a grouping of crystal structures according to the axial system used to describe their lattice. Each crystal system consists of a set of three axes in a particular geometrical arrangement. There are seven unique crystal systems. The simplest and most symmetric, the cubic (or isometric) system, has the symmetry of a cube, that is, it exhibits four threefold rotational axes oriented at 109.5 degrees (the tetrahedral angle) with respect to each other. These threefold axes lie along the body diagonals of the cube. This definition of a cubic is correct, although many textbooks
incorrectly state that a cube is defined by three mutually perpendicular axes of equal length – if this were true there would be far more than 14 Bravais lattices. The other six systems, in order of decreasing symmetry, are hexagonal, tetragonal, rhombohedral (also known as trigonal), orthorhombic, monoclinic and triclinic. Some crystallographers consider the hexagonal crystal system not to be its own crystal system, but instead a part of the trigonal crystal system. The crystal system and Bravais lattice of a crystal describe the (purely) translational symmetry of the crystal.

1.6.1 Lattice Constant:

The Lattice Constant [or lattice parameter] refers to the constant distance between unit cells in a crystal lattice. Lattices in three dimensions generally have three lattice constants, referred to as \( a \), \( b \), and \( c \). However, in the special case of cubic crystal structures, all of the constants are equal and we only refer to \( a \). Similarly, in hexagonal crystal structures, the \( a \) and \( b \) constants are equal, and we only refer to the \( a \) and \( c \) constants. A group of lattice constants could be referred to as lattice parameters. However, the full set of lattice parameters consist of the three lattice constants and the three angles between them.

For example the lattice constant for a common carbon diamond is \( a = 3.57 \text{Å} \) at 300 K. The structure is equilateral although its actual shape can not be determined from only the lattice constant. Furthermore, in real applications, typically the average lattice constant is given. As lattice constants have the dimension of length, their SI unit is the meter. Lattice constants are typically on the order of several angstroms. Lattice constants can be determined techniques such as X-ray diffraction.

In epitaxial growth, the lattice constant is a measure of the structural compatibility between different materials. Lattice constant matching is important for growth of thin layers of materials on other materials; when the
constants differ, strains are introduced into the layer, which prevents epitaxial growth of thicker layers without defects.

1.6.2 Bravais lattices in 2D:

In two dimensions, there are five Bravais lattices. They are oblique, rectangular, centered rectangular, hexagonal, and square, as shown in Figure (1.2).

![Bravais lattices in 2D](image)

**Fig.(1.2):** Bravais lattices in two dimensions: (1) oblique, (2) rectangular, (3) centered rectangular, (4) hexagonal, and (5) square.

In geometry and crystallography, a Bravais lattice is a category of symmetry groups for translational symmetry in three directions, or correspondingly, a category of translation lattices.

Such symmetry groups consist of translations by vectors of the form

$$R = n_1 a_1 + n_2 a_2 + n_3 a_3$$  \hspace{1cm} (1.1)

Where \( n_1, n_2, \text{ and } n_3 \) are integers and \( a_1, a_2, \text{ and } a_3 \) are three non-coplanar vectors, called primitive vectors.

These lattices are classified by space group of the translation lattice itself; there are 14 Bravais lattices in three dimensions; each can apply in one
crystal system only. They represent the maximum symmetry a structure with the translational symmetry concerned can have.

The Bravais lattices were studied by Moritz Ludwig Frankenheim (1801-1869), in 1842, who found that there were 15 Bravais lattices in three dimensions; this was corrected to 14 by A. Bravais in 1848.

1.6.3 The Bravais lattices in 3D:

When the crystal systems are combined with the various possible lattices centering, we arrive at the Bravais lattices. They describe the geometric arrangement of the lattice points, and thereby the translational symmetry of the crystal. In three dimensions, there are 14 unique Bravais lattices which are distinct from one another in the translational symmetry they contain. All crystalline materials recognized until now (not including quasicrystals) fit in one of these arrangements. The fourteen three-dimensional lattices, classified by crystal system, are shown to the Figure (1.3). The Bravais lattices are sometimes referred to as space lattices.

The crystal structure consists of the same group of atoms, the basis, positioned around each and every lattice point. This group of atoms therefore repeats indefinitely in three dimensions according to the arrangement of one of the 14 Bravais lattices. The characteristic rotation and mirror symmetries of the group of atoms, or unit cell, are described by its crystallographic point group.
<table>
<thead>
<tr>
<th>The 7 Crystal systems</th>
<th>The 14 Bravais Lattices:</th>
</tr>
</thead>
<tbody>
<tr>
<td>triclinic</td>
<td><img src="image1.png" alt="Image" /></td>
</tr>
<tr>
<td>monoclinic</td>
<td><img src="image2.png" alt="Image" /></td>
</tr>
<tr>
<td>orthorhombic</td>
<td><img src="image3.png" alt="Image" /></td>
</tr>
<tr>
<td>hexagonal</td>
<td><img src="image4.png" alt="Image" /></td>
</tr>
<tr>
<td>rhombohedral</td>
<td><img src="image5.png" alt="Image" /></td>
</tr>
<tr>
<td>tetragonal</td>
<td><img src="image6.png" alt="Image" /></td>
</tr>
<tr>
<td>cubic</td>
<td><img src="image7.png" alt="Image" /></td>
</tr>
</tbody>
</table>

**Fig. (1.3):** The 14 Bravais lattices in three dimension.
There are seven crystal systems:

1. **Triclinic**, all cases not satisfying the requirements of any other system. There is no necessary symmetry other than translational symmetry, although inversion is possible.

2. **Monoclinic**, requires either 1 twofold axis of rotation or 1 mirror plane.

3. **Orthorhombic**, requires either 3 twofold axes of rotation or 1 twofold axis of rotation and two mirror planes.

4. **Tetragonal**, requires 1 fourfold axis of rotation.

5. **Rhombohedral**, also called trigonal, requires 1 threefold axis of rotation.

6. **Hexagonal**, requires 1 six fold axis of rotation.

7. **Cubic or Isometric**, requires 4 threefold axes of rotation.

The table (1.1) gives a brief characterization of the various crystal systems. There are 2, 13, 59, 68, 25, 27, and 36 space groups per crystal system, respectively, for a total of 230.

*Table (1.1): Characterization of the various crystal system.*

<table>
<thead>
<tr>
<th>Crystal system</th>
<th>No. of point groups</th>
<th>No. of bravais lattices</th>
<th>No. of space groups</th>
</tr>
</thead>
<tbody>
<tr>
<td>Triclinic</td>
<td>2</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>Monoclinic</td>
<td>3</td>
<td>2</td>
<td>13</td>
</tr>
<tr>
<td>Orthorhombic</td>
<td>3</td>
<td>4</td>
<td>59</td>
</tr>
<tr>
<td>Tetragonal</td>
<td>7</td>
<td>2</td>
<td>68</td>
</tr>
<tr>
<td>Rhombohedral</td>
<td>5</td>
<td>1</td>
<td>25</td>
</tr>
<tr>
<td>Hexagonal</td>
<td>7</td>
<td>1</td>
<td>27</td>
</tr>
<tr>
<td>Cubic</td>
<td>5</td>
<td>3</td>
<td>36</td>
</tr>
<tr>
<td>Total</td>
<td>32</td>
<td>14</td>
<td>230</td>
</tr>
</tbody>
</table>
1.7 Point and space groups:

The crystallographic point group or crystal class is the mathematical group comprising the symmetry operations that leave at least one point unmoved and that leave the appearance of the crystal structure unchanged. These symmetry operations can include reflection, which reflects the structure across a reflection plane, rotation, which rotates the structure a specified portion of a circle about a rotation axis, inversion which changes the sign of the coordinate of each point with respect to a center of symmetry or inversion point and improper rotation, which consists of a rotation about an axis followed by an inversion. Rotation axes (proper and improper), reflection planes, and centers of symmetry are collectively called symmetry elements. There are 32 possible crystal classes. Each one can be classified into one of the seven crystal systems.

The space group of the crystal structure is composed of the translational symmetry operations in addition to the operations of the point group. These include pure translations which move a point along a vector, screw axes, which rotate a point around an axis while translating parallel to the axis, and glide planes, which reflect a point through a plane while translating it parallel to the plane. There are 230 distinct space groups.

1.8 Miller index:

Miller indices are a notation system in crystallography for planes and directions in crystal (Bravais) lattices.

In particular, a family of lattice planes is determined by three integers $h$, $k$, and $l$, the Miller indices. They are written $(hkl)$ and denote planes orthogonal to a direction $(h,k,l)$ in the basis of the reciprocal lattice vectors. By convention, negative integers are written with a bar, as in $\overline{3}$ for $-3$. The integers are usually written in lowest terms, i.e. their greatest common divisor should be $l$. 
There are also several related notations. \([hkl]\), with square instead of round brackets, denotes a direction in the basis of the direct lattice vectors instead of the reciprocal lattice. The notation \(\{hkl\}\) denotes all planes that are equivalent to \((hkl)\) by the symmetry of the crystal. Similarly, the notation \(\langle hkl \rangle\) denotes all directions that are equivalent to \([hkl]\) by symmetry.

Miller indices were introduced in 1839 by the British mineralogist William Hallows Miller. The method was also historically known as the Millerian system, and the indices as Millerian, although this is now rare.

Some of important planes in a cubic crystal structure are shown in Figure (1.4), while Figure (1.5) shows different direction in crystal structure.

\[\begin{align*}
\text{Fig.(1.4): Planes with different Miller indices in cubic crystals.} \\
\text{Fig.(1.5): Examples of directions in crystal structure.}
\end{align*}\]
The precise meaning of this notation depends upon a choice of lattice vectors for the crystal, as described below. Usually, three primitive lattice vectors are used. However, for cubic crystal systems, the cubic lattice vectors are used even when they are not primitive (e.g., as in body-centered and face-centered crystals). Figure (1.6) shows plane of (111) and (221) for non cubic crystal.

\[ \text{Fig.(1.6): Examples of determining indices for a plane using intercepts with axes; left (111), right (221).} \]

There are two equivalent ways to define the meaning of the Miller indices: via a point in the reciprocal lattice, or as the inverse intercepts along the lattice vectors. Both definitions are given below. In either case, one needs to choose the three lattice vectors \( \mathbf{a}_1, \mathbf{a}_2, \) and \( \mathbf{a}_3 \) as described above. Given these, the three primitive reciprocal lattice vectors are also determined (denoted \( \mathbf{b}_1, \mathbf{b}_2, \) and \( \mathbf{b}_3 \)).

Then, given the three Miller indices \( h,k,l \), \( (hkl) \) denotes planes orthogonal to:

\[ lb_1 + mb_2 + nb_3 \]

That is, \( (hkl) \) simply indicates a normal to the planes in the basis of the primitive reciprocal lattice vectors. Because the coordinates are integers, this normal is itself always a reciprocal lattice vector. The requirement of lowest terms means that it is the shortest reciprocal lattice vector in the given direction.
Equivalently, \((hkl)\) denotes a plane that intercepts the three points \(\frac{a_1}{h}, \frac{a_2}{k}, \) and \(\frac{a_3}{l}\), or some multiple thereof. That is, the Miller indices are proportional to the inverses of the intercepts of the plane, in the basis of the lattice vectors. If one of the indices is zero, it means that the planes do not intersect that axis (the intercept is "at infinity").

The related notation \([hkl]\) denotes the direction:

\[la_1 + ma_2 + na_3\]

That is, it uses the direct lattice basis instead of the reciprocal lattice. Note that \([hkl]\) is not generally normal to the \((hkl)\) planes, except in a cubic lattice as described below.

For the special case of simple cubic crystals, the lattice vectors are orthogonal and of equal length; similarly for the reciprocal lattice. So, in this common case, the Miller indices \((hkl)\) and \([hkl]\) both simply denote normals directions in Cartesian coordinates.

For face-centered cubic and body-centered cubic lattices, the primitive lattice vectors are not orthogonal. However, in these cases the Miller indices are conventionally defined relative to the lattice vectors of the cubic super cell and hence are again simply the Cartesian directions.

1.9 Miller-Bravais index:

With hexagonal and rhombohedral crystal systems, it is possible to use the Bravais-Miller index which has 4 numbers \((h k i l)\),

\[i = -h - k.\]

Here \(h, k\) and \(l\) are identical to the Miller index, and \(i\) is a redundant index.

This four-index scheme for labeling planes in a hexagonal lattice makes permutation symmetries apparent. For example, the similarity between \((110) \equiv (11\bar{2}0)\) and \((1\bar{2}0) \equiv (1\bar{2}10)\) is more obvious when the redundant index is shown.

In the figure (1.7), the \((001)\) plane has a 3-fold symmetry: it remains unchanged by a rotation of \(1/3\) \((2\pi/3 \text{ rad}, 120^\circ)\). The \([100], [010]\) and the
[\overline{1}\overline{1}0] directions are really similar. If $S$ is the intercept of the plane with the [1\overline{1}0] axis, then

$$i = 1/S.$$ 

![Diagram](image)

**Fig.(1.7):** Miller-Bravis index of hexagonal and rhombohedral structures.

There are also reciprocal lattice vectors or planes, for indexing hexagonal lattice vectors with four indices. However they don't operate by similarly adding a redundant index to the regular three-index set.

For example, the reciprocal lattice vector $(hkl)$ as suggested above can be written as $ha^*+kb^*+lc^*$ if the reciprocal-lattice basis-vectors are $a^*$, $b^*$, and $c^*$. For hexagonal crystals this may be expressed in terms of direct-lattice basis-vectors $a$, $b$ and $c$ as:

$$(hkl) = h\overrightarrow{a^*} + k\overrightarrow{b^*} + l\overrightarrow{c^*} = \frac{2}{3a^2}(2h + k)\overrightarrow{a} + \frac{2}{3a^2}(h + 2k)\overrightarrow{b} + \frac{1}{c^2}(l)\overrightarrow{c}$$

Hence zone indices of the direction perpendicular to plane $(hkl)$ are, in suitably-normalized triplet form, simply $[2h+k,h+2k,l(3/2)(a/c)^2]$. When four indices are used for the zone normal to plane $(hkl)$, however, the literature often uses $[h, k,-h-k, l (3/2) (a/c)^2]$ instead. Thus as you can see, four-index zone indices in square or angle brackets sometimes mix a single direct-lattice index on the right with reciprocal-lattice indices (normally in round or curly brackets) on the left.

Ordinarily, Miller indices are always integers by definition, and this constraint is physically significant. To understand this, suppose that we allow...
a plane \((ABC)\) where the Miller "indices" \(a, b,\) and \(c\) (defined as above) are not necessarily integers.

If \(a, b,\) and \(c\) have rational ratios, then the same family of planes can be written in terms of integer indices \((hkl)\) by scaling \(a, b,\) and \(c\) appropriately: divide by the largest of the three numbers, and then multiply by the least common denominator. Thus, integer Miller indices implicitly include indices with all rational ratios.
Chapter Two

X-ray diffraction Analysis

2.1 Introduction:

After our preliminary survey of the geometry of crystals, we can now proceed to discuss the phenomenon of x-ray diffraction, which is an interaction of the X-ray with material. Historically, this is exactly the way this field of science developed. For many years, mineralogists and crystallographers had accumulated knowledge about crystals, chiefly by measurement of interfacial angles, chemical analysis, and determination of physical properties. There was little knowledge of interior structure, however, although some very shrewd guesses had been made, namely, that crystal; were built up by periodic repetition of some unit, probably an atom or molecule, and that these units were situated some 1 or 2 Å apart. On the other hand, there were indications, but only indications, that x-rays might be electromagnetic waves about 1 or 2 Å in wavelength. In addition, the phenomenon of diffraction was well understood, and it was known that diffraction, as of visible light by a ruled grating, occurred whenever wave motion encountered a set of regularly spaced scattering objects, provided that the wavelength of the wave motion was of the same order of magnitude as the repeat distance between the scattering centers.

2.2 The Bragg diffraction law:

The Bragg's law is the result of experiments into the diffraction of X-rays or neutrons off crystal surfaces at certain angles, derived by physicist Sir William Lawrence Bragg in 1912. Although simple, Bragg's law confirmed the existence of real particles at the atomic scale, as well as providing a powerful new tool for studying crystals in the form of X-ray and neutron diffraction. William Lawrence Bragg and his father, Sir William Henry
Bragg, were awarded the Nobel Prize in physics in 1915 for their work in determining crystal structures beginning with NaCl, ZnS, and diamond.

When X-rays hit an atom, they make the electronic cloud move as does any electromagnetic wave. The movement of these charges re-radiates waves with the same frequency see Figure (2.1) this phenomenon is known as the Raleigh scattering (or elastic scattering). The scattered waves can themselves be scattered but this secondary scattering is assumed to be negligible. A similar process occurs upon scattering neutron waves from the nuclei or by a coherent spin interaction with an unpaired electron. These re-emitted wave fields interfere with each other either constructively or destructively (overlapping waves either add together to produce stronger peaks or subtract from each other to some degree), producing a diffraction pattern on a detector or film [see Figure (2.2)]. The resulting wave interference pattern is the basis of diffraction analysis. Both neutron and X-ray wavelengths are comparable with inter-atomic distances (~150 pm) and thus are an excellent probe for this length scale.

\[ n\lambda = 2d \cdot \sin \theta \]

Where
- \( n \) is an integer determined by the order given,
- \( \lambda \) is the wavelength of x-rays, and moving electrons, protons and neutrons,
- \( d \) is the spacing between the planes in the atomic lattice, and
- \( \theta \) is the angle between the incident ray and the scattering planes.
According to the $2\theta$ deviation, the phase shift causes constructive (left figure) or destructive (right figure) interferences.

2.3 The reciprocal lattice:

In crystallography, the reciprocal lattice of a Bravais lattice is the set of all vectors $K$ such that:

$$e^{iK\cdot R} = 1$$

For all lattice point position vectors $R$. This reciprocal lattice is itself a Bravais lattice, and the reciprocal of the reciprocal lattice is the original lattice.

For an infinite three dimensional lattice, defined by its primitive vectors $(a_1, a_2, a_3)$, its reciprocal lattice can be determined by generating its three reciprocal primitive vectors. The simple cubic Bravais lattice, with cubic primitive cell of side $a$, has for its reciprocal a simple cubic lattice with a cubic primitive cell of side $\frac{2\pi}{a} \left( \frac{1}{a} \right)$ in the crystallographer's definition). The cubic lattice is therefore said to be dual, having its reciprocal lattice being identical (up to a numerical factor).

The reciprocal lattice to a BCC lattice is the FCC lattice. It can be easily proven that only the Bravais lattices which have 90 degrees between $(a_1, a_2, a_3)$ (cubic, tetragonal, orthorhombic) have $(b_1, b_2, b_3)$ parallel to their real-space vectors.

It is interesting to consider this vector and its components in some detail, in view of its numerous applications in crystallography.

Let us first rewrite $G$ as

$$G = h \mathbf{a}^* + k \mathbf{b}^* + l \mathbf{c}^* \quad (2.1)$$
Where

The reciprocal lattice

\[ \mathbf{a}^* = (\mathbf{b} \times \mathbf{c})/V \] (2.2)
\[ \mathbf{b}^* = (\mathbf{c} \times \mathbf{a})/V \] (2.3)
\[ \mathbf{c}^* = (\mathbf{a} \times \mathbf{b})/V \] (2.4)

and \( V \) is the volume of the unit cell.

Let us now allow \( hkl \) to take on any integer value that is we shall not restrict them to relatively prime integers. The vector \( \mathbf{G} \) now becomes a linear combination of the basis vectors \( \mathbf{a}^* \), \( \mathbf{b}^* \), and \( \mathbf{c}^* \), the coefficients of this combination being any integers. Mathematically, this means that \( \mathbf{G} \) is a lattice vector, these two lattices are different and its basis vector \( \mathbf{a} \), \( \mathbf{b} \), and \( \mathbf{c} \) has dimensions of length, while \( \mathbf{G} \) and its basis vectors \( \mathbf{a}^*, \mathbf{b}^*, \) and \( \mathbf{c}^* \) have dimensions length\(^{-1}\). For this reason, as well as others which will become clear later, we call the set of all the points with position vectors.

\[ \mathbf{r} = u\mathbf{a} + v\mathbf{b} + w\mathbf{c} \] (2.5)

Where \( uvw \) are any integers, the direct lattice, and the set of all the points with position vectors

\[ \mathbf{G} = h\mathbf{a}^* + k\mathbf{b}^* + l\mathbf{c}^* \] (2.6)

Where \( hkl \) are any integers, the reciprocal lattice.

We shall now proceed to inspect the relationships between the bases of the direct and reciprocal lattices. It is evident from that \( \mathbf{a}^* \) is perpendicular to \( \mathbf{b} \) and to \( \mathbf{c} \), \( \mathbf{b}^* \) is perpendicular to \( \mathbf{c} \) and to \( \mathbf{a} \), and \( \mathbf{c}^* \) is perpendicular to \( \mathbf{a} \) and to \( \mathbf{b} \). We therefore have

\[ \mathbf{a}^* \cdot \mathbf{b} = \mathbf{a}^* \cdot \mathbf{c} = \mathbf{b}^* \cdot \mathbf{c} = \mathbf{b}^* \cdot \mathbf{a} = \mathbf{c}^* \cdot \mathbf{a} = \mathbf{c}^* \cdot \mathbf{b} = 0 \] (2.7)

We further have

\[ \mathbf{a} \cdot \mathbf{a}^* = (\mathbf{b} \times \mathbf{c})/V = V/V = 1 \] (2.8)

and similarly for the scalar products involving \( \mathbf{b} \) and \( \mathbf{c} \). That is:

\[ \mathbf{a} \cdot \mathbf{a}^* = \mathbf{b} \cdot \mathbf{b}^* = \mathbf{c} \cdot \mathbf{c}^* = 1 \] (2.9)

Indicates further that: \( \mathbf{a}^* \cdot (\mathbf{b}^* \times \mathbf{c}) \), \( \mathbf{b}^* \cdot (\mathbf{c}^* \times \mathbf{a}^*) \) and \( \mathbf{c}^* \cdot (\mathbf{a}^* \times \mathbf{b}^*) \).

If we combine this result with, we obtain
\[ a = \frac{(b \times c)}{V} \quad (2.10) \]
\[ b = \frac{(c \times a)}{V} \quad (2.11) \]
\[ c = \frac{(a \times b)}{V} \quad (2.12) \]

Where
\[ V^* = a^* \cdot (b^* \times c^*) = b^* \cdot (c^* \times a^*) = c^* \cdot (a^* \times b^*) \]
is the volume of the unit cell of the reciprocal lattice, based on the vectors \( a^* \), \( b^* \) and \( c^* \).

Note further that also
\[ V^* = a^* \cdot (b^* \times c^*) = (b \times c) \cdot (b^* \times c^*) / V \]
\[ = (b \cdot b^*) \cdot (c \cdot c^*) - (b \cdot c^*) \cdot (c \cdot b^*)V \]
\[ = 1/V \quad (2.13) \]

That is, the volumes of the direct-lattice and reciprocal-lattice unit cells also turn out to be mutually reciprocal. Here use has been made of the standard vector-algebraic expression for a scalar product of two vector products.

Let us now find an expression for \( V \) in terms of the unit-cell parameters \( a, b, c, \alpha, \beta, \gamma \). A straightforward approach to the calculation of the volume of a parallelepiped in terms of its edges and angles is rather cumbersome, and we shall follow the method of Buerger (1941) which is far simpler. If we refer the basis vectors \( a, b, \) and \( c \) to a Cartesian system, say
\[ a = iax + jay + kaz \quad (2.14) \]
\[ b = ibx + jby + kbx \quad (2.15) \]
\[ c = icx + jcy + kcz \quad (2.16) \]

2.4 Ewald Construction:

P. P. Ewald was responsible for interpreting Laue's results in terms of reciprocal lattices. He devised a simple geometric construction that demonstrates the relationship in quite elegant but simple terms.

He separated the origins of direct and reciprocal space placing the crystal at the center of a sphere of radius \( 1/l \). The origin of reciprocal space is placed
on the surface of the sphere where $S_0$ (the incident beam) exits the sphere, as shown in Figure (2.3).

![Diagram](image)

**Fig.(2.3): Ewald Construction.**

A highly suggestive geometrical description of the conditions for direction was put forward by Ewald (1913), and is employed very extensively in the crystallographic literature, albeit in several dieting representations. Let us resend the maximum value of $|G|$.

$$|G| = 1dhkl = 2\lambda \sin \theta$$  \hspace{1cm} (2.17)

And since the maximum value of $\sin \theta$ is 1, we must have $|G|_{\text{max}} = 2\lambda$.

### 2.5 Experiment X-ray diffraction method:

A large range of laboratory equipment is available for x-ray diffraction and spectroscopy, and the International Union of Crystallography has published a useful, which shows what apparatus and supplies are available and where to find them. A laboratory manual by Azaroff and Donahue describes twenty-one experiments in x-ray crystallography. This section deals with some experimental methods to find crystal structure by x-ray diffraction.
2.5.1 The Laue method:

Laue photographs are the easiest kind of diffraction pattern to make and require only the simplest kind of apparatus. White radiation is necessary, and the best source is a tube with a heavy-metal target, such as tungsten, since the intensity of the continuous spectrum is proportional to the atomic number of the target metal. Good patterns can also be obtained with radiation from other metals, such as molybdenum or copper.

The specimen used in the Laue method is a single crystal. This may mean an isolated single crystal or one particular crystal grain, not too small, in a polycrystalline aggregate. The only restriction on the size of a crystal in a polycrystalline mass is that it must be no smaller than the incident x-ray beam, if the pattern obtained is to correspond to that crystal alone.

Unlike the other data collection methods, the crystal is kept stationary during Laue data collection. Instead, reflections are made to intersect the Ewald sphere by changing the wavelength. As the wavelength changes the radius of the Ewald sphere changes as well. Changing the wavelength continuously has been carried out, but normally the crystals are exposed to "white radiation". This means that the synchrotron radiation is not monochromatic ed so that the full wavelength band path illuminates the crystal. Filters to attenuate long-wavelength radiation may be used to remove X-rays that are not useful but do heat up the crystal and cause radiation damage. Figure (2.4) shows the Laue diffraction geometry as a Ewald sphere representation.
Fig.(2.4): In a Laue experiment, X-rays of all wavelengths in a certain wavelength range (Lambda-min to Lambda-max) are present.

Each wavelength corresponds to a Ewald sphere with a different radius. All of these will fall between the spheres of the maximum and minimum wavelength (shown in gray). All reflections in the gray area that fall within the limiting sphere will diffract simultaneously.

The strength of the Laue experiment is that a very large fraction of the reciprocal lattice diffracts simultaneously. This means that a large fraction of the unique data can be collected on one or just a few images. This strength is also a weakness because the large number of simultaneously diffracting reflections means that a significant fraction of them will be spatially overlapped. Data collection is also more complicated as corrections have to be made for reflections collected at different wavelengths. Finally, all wavelengths contribute to X-ray background scatter whereas only a very narrow wavelength range leads to diffraction of each reflection. Therefore, the background is much higher than necessary, leading to a reduced signal to noise. As a consequence, Laue diffraction is not a general purpose technique and should only be used if your scientific questions require very rapid data collection.
The diffracted beams form arrays of spots that lie on curves on the film. The Bragg angle is fixed for every set of planes in the crystal. Each set of planes picks out and diffracts the particular wavelength from the white radiation that satisfies the Bragg law for the values of $d$ involved. Each curve therefore corresponds to a different wavelength. The spots lying on any one curve are reflections from planes belonging to one zone. Laue reflections from planes of the same zone all lie on the surface of an imaginary cone whose axis is the zone axis.

There are two practical variants of the Laue method, the back-reflection and the transmission Laue method.

1- **Back-reflection Laue:**

In the back-reflection method, the film is placed between the X-ray source and the crystal. The beams which are diffracted in a backward direction are recorded. One side of the cone of Laue reflections is defined by the transmitted beam. The film intersects the cone, with the diffraction spots generally lying on an hyperbola, as shown in Figure (2.5).

![Laue Method](image)

*Fig.(2.5): Back-Reflection Laue Method.*

2- **Transmission Laue:**

In the transmission Laue method, the film is placed behind the crystal to record beams which are transmitted through the crystal. One side of the cone of Laue reflections is defined by the transmitted beam. The film intersects the
cone, with the diffraction spots generally lying on an ellipse, as illustrated in Figure (2.6).

Fig.(2.6): Transmission Laue Method.

2.5.2 Rotating-crystal method:

In the rotating-crystal method a single crystal is mounted with one of its axes, or some important crystallographic direction, normal to a monochromatic x-ray beam. A cylindrical film is placed around it and the crystal is rotated about the chosen direction, the axis of the film coinciding with the axis of rotation of the crystal (Figure (2-7)). As the crystal rotates, a particular set of lattice planes will, for an instant, make the correct Bragg angle for reflection of the monochromatic incident beam, and at that instant a reflected beam will be formed. The reflected beams are again located on imaginary cones but now the cone axes coincide with the rotation axis. The result is that the spots on the film, when, the film is laid out flat, lie on imaginary horizontal lines, as shown in Figure (2.8). Since the crystal is rotated about only one axis, the Bragg angle does not take on all possible values between 0° and 90° for every set of planes. Not every set, therefore, is able to produce a diffracted beam; sets perpendicular or almost perpendicular to the rotation axis are examples.

The chief use of the rotating-crystal method and its variations is in the determination of unknown crystal structures, and for this purpose it is the most powerful tool the x-ray crystallographer has at his disposal. However, the
complete determination of complex crystal structures is a subject beyond the scope of this book and outside the province of the average metallurgist who uses x-ray diffraction as a laboratory tool.

**Fig.(2-7):** Rotating-crystal methods.

**Fig.(2.8):** Rotating-crystal pattern of a quartz crystal (hexagonal) rotated about its c axis. Filtered copper radiation.

When monochromatic radiation is incident on a single crystal rotated about one of its axes, the reflected beams lie on the surface of imaginary cones coaxial with the rotation axis. The way in which this reflection occurs may be shown very nicely by the Ewald construction. Suppose a simple cubic crystal is rotated about the axis [001]. This is equivalent to rotation of the reciprocal lattice about the b\textsubscript{3} axis. Figure (2.9) shows a portion of the reciprocal lattice oriented in this manner, together with the adjacent sphere of reflection. All crystal planes having indices (Mi) are represented by points lying on a plane in the reciprocal lattice, normal to b\textsubscript{3}. When the reciprocal lattice rotates this plane cuts the reflection sphere in the small "circle"
shown, and any points on the layer which touch the sphere surface must touch it on this circle. Therefore all diffracted-beam vectors must end on this circle, which is equivalent to saying that the diffracted beams must lie on the surface of a cone. In this particular case, all the $hkl$ points shown intersect the surface of the sphere sometime during their rotation about the $b3$ axis, producing the diffracted beams shown in Figure (2-9).

This simple example may suggest how the rotations photograph of a crystal of unknown structure, and therefore having an unknown reciprocal lattice, can yield clues as to the distribution in space of reciprocal-lattice points. By taking a number of photographs with the crystal rotated successively about various axes, the crystallographer gradually discovers the complete distribution of reflecting points. Once the reciprocal lattice is known, the crystal lattice is easily derived.

![Figure (2.9): Reciprocal-lattice treatment of rotating-crystal method.](image)

### 2.5.3 The powder method:

Rather than using white radiation, take monochromatic X-radiation of one fixed wavelength and place the crystal in front of the beam. If one plane is set at exactly the correct value of $\theta$ to reflect, then we observe one and only one
reflected beam from that crystal. Imagine now, still holding the crystal fixed at the angle $\theta$, we rotate the crystal around the direction of the incident X-ray beam so that the plane causing a reflection is still set at the angle $\theta$ relative to the X-ray beam. The reflected beam will describe a cone with the crystal at the apex of the cone. Now imagine the situation when we have not one crystal but we have a hundred crystals each of them set so that one plane is at exactly the right reflecting angle, to the incoming beam. We will now have a hundred reflected beams each giving us one observable point. Imagine now this clump of a hundred crystals is rotated about the axis of the incident X-ray beam. We will now have a hundred cones traced out by these reflected beams. Now consider what would happen if we had a powder of our material which may consist of a hundred million crystals. If the powdered sample is put into the beam of X-rays, there will be many crystals in that powder which will be in a position to reflect the incident beam and there will be enough of them to get the effect of not point reflections but of a continuous series of point reflections which will be lying along the arc of the cone that we previously imagined to exist. This is the basis of the so-called powder or Debye-Scherrer method which is probably the most common technique used in X-ray crystallography. Figure (2.10) shows a schematic draw of this method.

![Schematic of Debye-Scherrer Method](image)

**Fig.(2.10):** Schematic of Debye-Scherrer Method.
The powder camera consists of a metal cylinder at the centre of which is the sample. The powdered material is often glued onto a glass rod with clear fingernail varnish. A strip of X-ray film is placed inside the cylinder. Punched into one side of the film is a hole for the beam collimator and punched into the other side 180° away, is another hole through which a beam catcher can be placed. The camera is closed by a light-tight lid and placed in front of the X-ray beam. The pattern on the film is shown in Figure (2.11)

This technique can be adapted to photographing wires and sheets of metal. A flat film is also commonly used for recording reflections at small θ angles.

**Fig.(2.11): X-ray crystallography film.**

a) Photography Technique:

Photographic film is affected by x-rays in much the same way as by visible light. However, the emulsion on ordinary film is too thin to absorb much of the incident x-radiation, and only absorbed x-rays can be effective in blackening the film. For this reason, x-ray films are made with rather thick layers of emulsion on both sides in order to increase the total absorption. (Division of the total emulsion thickness into two layers permits easier
penetration of the film-processing solutions.) The grain size is also made large for the same purpose: this has the unfortunate consequence that x-ray films are grainy, do not resolve fine detail, and cannot stand much enlargement.

Because the mass absorption coefficient of any substance varies with wavelength, it follows that film sensitivity, i.e., the amount of blackening caused by x-ray beams of the same intensity, depends on their wavelength. This should be borne in mind whenever white radiation is recorded photographically; for one thing, this sensitivity variation alters the effective shape of the continuous spectrum.

b) Diffractometer technique:

The x-ray spectrometer can also be used as a tool in diffraction analysis. This instrument is known as a diffractometer when it is used with x-rays of know wavelength to determine the unknown spacing of crystal planes, and as a spectrometer in the reverse case, when crystal planes of known spacing are used to determine unknown wavelengths. The diffractometer is always used with monochromatic radiation and measurements may be’ made oh either single crystals or poll crystal-line specimens; in the latter case, it functions much like a Debye-Scherrer camera in that the counter intercepts and measures only a short arc of any one cone of diffracted rays.

2.6 Geometrical structure factor:

If the crystal structure is that of a monatomic lattice with an n-atom basis, then the contents of each primitive cell can be analyzed into a set of identical scatters at positions r_1, r_2, ... r_n within the cell. The intensity of radiation in a given Bragg peak will depend on the extent to which the rays scattered from these basis sites interfere with one another, being greatest when there is complete constructive interference and vanishing altogether should there happen to be complete destructive interference. The net ray scattered by the
entire primitive cell is the sum of the individual rays, and will therefore have amplitude containing the factor:

\[ S_G = \sum_{j=1}^{n} e^{i\mathbf{G} \cdot \mathbf{r}_j} \]  

(2.18)

where \( G = (h \mathbf{a^*}, k \mathbf{b^*}, l \mathbf{c^*}) \); \( \mathbf{a^*}, \mathbf{b^*}, \mathbf{c^*} \) is the unit vector of a reciprocal lattice.

The quantity \( S_G \), known as the geometrical structure factor, expresses the extent to which interference of the waves scattered from identical ions within the basis can diminish the intensity of the Bragg peak associated with the reciprocal lattice vector \( G \). The intensity in the Bragg peak, being proportional to the square of the absolute value of the amplitude, will contain a factor \( |S_G|^2 \). It is important to note that this is not the only source of \( G \) dependence in the intensity. Further dependence on the change in wave vector comes both from the ordinary angular dependence of any electromagnetic scattering, together with the influence on the scattering of the detailed internal structure of each individual ion in the basis. Therefore, the structure factor alone cannot be used to predict the absolute intensity in a Bragg peak. It can however, lead to a characteristic dependence on \( G \) that is easily discerned even though other less distinctive \( G \) dependences have been superimposed upon it. The one case in which the structure factor can be used with assurance is when it vanishes. This occurs when the elements of the basis are so arranged that there is complete destructive interference for the \( G \) in question. For each of the possible space groups there are characteristic absences of reflections, and from these the space group is determined.

The missing diffraction line for simple cubic, body-centered cubic, face-centered cubic, and diamond are illustrated in Figure (2.12).
\textbf{Fig. (2.12):} Powder patterns for different cubic crystals, illustrating characteristic reflections and absences for each type.

2.7 Atomic Form Factor:

If the ions in the basis are not identical, the structure factor assumes the form:

\[ S_G = \sum_{j=1}^{n} f_j e^{i \vec{G} \cdot \vec{r}_j} \]  \hspace{1cm} (2.19)

Where \( f_j \), known as the atomic form factor, is entirely determined by the internal structure of the ion that occupies position \( \vec{d}_j \) in the basis. This form factor is the ratio of the amplitude of the radiation scattered by the atom to the amplitude of the radiation which a single electron would scatter under the same conditions according to classical theory. In the non relativistic approximation the form factor is given by:

\[ f(s) = \int \psi^*_i(\vec{r}) \exp(i\vec{s} \cdot \vec{r}) \psi_i(\vec{r}) \, dv \]  \hspace{1cm} (2.20)

\( \psi \) is the total wave function of the atom and subscripts \( i \) and \( f \) refer to the initial and final states, \( \psi^* \psi \) is the charge distribution. The vector \( \vec{s} \) bisects the angle 180-20 between the incident and scattered wave vectors. The magnitude
of $s$ is $|s| = 4\pi \lambda^{-1} \sin \theta$. The vector $\lambda s/2\pi$ represents the change in momentum of the X-ray. If the charge density of the atom is spherically symmetric the form factor will be:

$$f(s) = \int_0^\infty 4\pi r^2 \rho(r) \frac{\sin sr}{sr} dr$$  \hspace{1cm} (2.21)

Note that $4\pi \int r^2 \rho(r) dr$ is equal to the total number of electrons $Z$ in the atom. Hence the atomic form factor is equal to $Z$ only for $\theta = 0$, and less than $Z$ for all other angles of scattering.

### 2.8 Structure-Factor Calculations:

When the diffraction condition $\Delta K = G$ is satisfied, the scattering amplitude (structure factor) is determined by:

$$S_k = \sum_{j=1}^{n} f_j e^{i\vec{G} \cdot \vec{r}_j}$$  \hspace{1cm} (2.22)

where $f_j$ is atomic scattering factor (form factor). The usual form of this result follows on writing, the lattice vector as:

$$\vec{r}_j = u_j \hat{a} + v_j \hat{b} + w_j \hat{c}$$  \hspace{1cm} (2.23)

Then, for the reflection labeled by $u_j, v_j, w_j$ (i.e. Reciprocal lattice vector), we have:

$$\vec{G} \cdot \vec{r}_j = 2\pi \left[ u_j h + v_j + w_j l \right]$$  \hspace{1cm} (2.24)

$$S_G = \sum_{j=1}^{n} f_j e^{2\pi i(u_j, h + v_j, k + w_j)}$$  \hspace{1cm} (2.25)

Where $n$ is a number of atom in unit cell, and $u_j, v_j, w_j$ was position of each atom in the unit cell.

Facility in the use of above equation can be gained only by working out some actual examples, and we shall consider a few such problems here.
a) **Structure Factor of the S.C. lattice:**

The basis of the S.C. structure referred to the cubic cell has identical one atom at \((0,0,0)\). Thus, equation (2.25) becomes:

\[ S_g = fe^{2\pi i(0)} = f \]

For any \(h, k, l\) thus in the S.C. all planes in the crystal are reflected, i.e. there are not any missing planes in the diffraction pattern.

b) **Structure Factor of the B.C.C. lattice:**

The basis of the B.C.C. structure referred to the cubic cell has two identical atoms at \((0,0,0)\) and \((1/2,1/2,1/2)\). i.e.

\[ u_1 = v_1 = w_1 = 0 \quad \& \quad u_2 = v_2 = w_2 = \frac{1}{2} \]

Substituting these values in equation (2.25):

\[ S_g = f_1 e^0 + f_2 e^{2\pi i \frac{1}{2}(h+k+l)} \]

\[ S_g = f_1 + f_2 e^{\pi i(h+k+l)} \quad \{e^{\theta} = \cos \theta + i \sin \theta\} \]

\[ S_g = f_1 + f_2 \{\cos[\pi(h+k+l)] + i \sin[\pi(h+k+l)]\} \]

For identical atoms \(f_1 = f_2\)

\[ S_g = f\{1 + \cos[\pi(h+k+l)] + i \sin[\pi(h+k+l)]\} \]

- if \(h+k+l = \text{odd integer}\) \(\Rightarrow S_g = 0\)
- if \(h+k+l = \text{even integer}\) \(\Rightarrow S_g = 2f\)

thus, in the B.C.C. lattice no reflections can occur for which the some of the indices is odd integers, the reflections occur only when the some of the indices is even integers. We had previously concluded from geometrical considerations that the base-centered cell would produce a 001 reflection but that the body-centered cell would not. This result is in agreement with the structure-factor equations for these two cells.
c) Structure Factor of the F.C.C. lattice:

The basis of the F.C.C. structure referred to the cubic cell has four identical atoms at the positions:

\[ 000, \frac{1}{2} 0 \frac{1}{2}, \frac{1}{2} 0 \frac{1}{2}, 0 \frac{1}{2} 0 \frac{1}{2} \]

Substituting these values in equation (2.25), we get:

\[ S_G = f \left[ e^{0} + e^{\pi i (h+k)} + e^{\pi i (h+l)} + e^{\pi i (k+l)} \right] \]

\[ S_G = f \left[ 1 + e^{\pi i (h+k)} + e^{\pi i (h+l)} + e^{\pi i (k+l)} \right] \]

* \[ S_G = 0 \] if \( h, k, l \) are mixed.

* \[ S_G = 4f \] if \( h, k, l \) are all even or all odd.

We conclude that, if all indices are even integers, \( S_G = 4f \); similarly if all indices are odd integers. But if only one of the integers is even, two of the exponents will be odd multiples of \(-i\pi\) and \( S_G \) will vanish. If only one of the integers is odd, the same argument applies and \( S_G \) will also vanish. Thus in the F.C.C. lattice no reflections can occur for which the indices are partly even and partly odd.
Chapter Three
Crystal Structure Determinations

3.1 Introduction:
When a material is irradiated with a parallel beam of monochromatic X-ray, the atomic lattice of the sample acts as a three dimensional diffraction grating causing the X-ray beam to be diffracted to specific angles. The diffraction pattern, that includes position (angles) and intensities of the diffracted beam, provides several information about the sample. Determination of indices (hkl) and unit cell (a) from powder scan is one of theses procedure.

For the sake of more understanding and practice our knowledge, the analyzed was done on the eight experimental spectrum of powder diffractometer produced by iron target X-ray tube that the characteristics wavelength of the incident X-ray was ($\lambda=0.193609 \, \text{nm}$). The entire spectrums are taken from “Crystal Structure Database for Inorganic Compounds”, Version 1.0.

3.2 Indexing patterns of cubic crystals:
A cubic crystal gives diffraction lines whose ($sin^2 \theta$) values satisfy the following equation, obtained by combining the Bragg law with the plane-spacing equation for the cubic system:

$$\frac{sin^2 \theta}{(h^2+k^2+l^2)} = \frac{sin^2 \theta}{s} = \frac{\lambda^2}{4a^2} \quad (3.1)$$

Since the sum $s = (h^2 + k^2 + l^2)$ is always integral and $\frac{\lambda^2}{4a^2}$ is a constant for any one pattern, the problem of indexing the pattern of a cubic substance is one of finding a set of integers (s) which will yield a constant quotient when divided one by one into the observed ($sin^2 \theta$) values. (Certain integers, such as 7, 15, 23, 28, 31, etc., are impossible because they cannot be
formed by the sum of three squared integers). Once the proper integers \(s\) are found, the indices \(hkl\) of each line can be written down by inspection. The proper set of integers \(s\) is not hard to find because there are only a few possible sets. Each of the four common cubic lattice types has a characteristic sequence of diffraction lines, described by their sequential \(s\) values:

- Simple cubic: \(1, 2, 3, 4, 5, 6, 8, 9, 10, 11, 12, 13, 14, 16,...\)
- Body-centered cubic: \(2, 4, 6, 8, 10, 12, 14, 16,...\)
- Face-centered cubic: \(3, 4, 8, 11, 12, 16,...\)
- Diamond cubic: \(3, 8, 11, 16,...\)

Each set can be tried in turn. If a set of integers satisfying Eq. (3.1) cannot be found, then the substance involved does not belong to the cubic system, and other possibilities (tetragonal, hexagonal, etc.) must be explored.

The following example will illustrate the steps involved in indexing the pattern of a cubic substance and finding its lattice parameter. In this particular example \(Fe\) \(\alpha\) radiation was used and eight diffraction lines were observed. Their \((\sin^2\theta)\) values are listed in the second column of Tables. After a few trials, the integers \(s\) listed in the third column were found to produce the reasonably constant quotients listed in the fourth column, when divided into the observed \((\sin^2\theta)\) values. The fifth column lists the lattice parameter calculated from each line position, and the sixth column gives the Miller indices of each line. The systematic error in \((\sin^2\theta)\) shows up as a gradual decrease in the value of \(\lambda^2/4a^2\), and a gradual increase in the value of \(a\), as \(\theta\) increases. In addition, the Bravais lattice of the specimen can be determined by observing which lines are present and which absent.

The calculations are made for \(Fe\) \(K\alpha\) radiation \((\lambda=0.193609\;nm)\). The positions of all the diffraction lines which would be formed under these conditions are indicated as they would appear on a film.

Powder patterns of cubic substances can usually be distinguished at a glance from those of noncubic substances, since the latter patterns normally
contain many more lines. In addition, the Bravais lattice can usually be identified by inspection: there is an almost regular sequence of lines in simple cubic and body-centered cubic patterns, but the former contains almost twice as many lines, while a face-centered cubic pattern is characterized by a pair of lines, followed by a single line, followed by a pair, another single line, etc.

The problem of indexing a cubic pattern is of course very much simplified if the substance involved is known to be cubic and if the lattice parameter is also known. The simplest procedure then is to calculate the value of \((\lambda^2/4a^2)\) and divide this value into the observed \((\sin^2\theta)\) values to obtain the value of \((s)\) for each line.

There is one difficulty that may arise in the interpretation of cubic powder patterns, and that is due to a possible ambiguity between simple cubic and body-centered cubic patterns. There is a regular sequence of lines in both patterns up to the sixth line; the sequence then continues regularly in body-centered cubic patterns, but is interrupted in simple cubic patterns since \((s = 7)\) is impossible. Therefore, if \(\lambda\) is so large, or \(a\) so small, that six lines or less appear on the pattern, the two Bravais lattices are indistinguishable. For example, suppose that the substance involved is actually body-centered cubic but the investigator mistakenly indexes it as simple cubic, assigning the value \((s = 1)\) to the first line, \((s = 2)\) to the second line, etc. He thus obtains a value of \((\lambda^2/4a^2)\) twice as large as the true one, and a value of \(a\) which is \((1/\sqrt{2})\) times the true one. This mistake becomes apparent when the number of atoms per unit cell is calculated from the measured density of the specimen; the wrong cell size will give a nonintegral value for the number of atoms per cell, and such a value is impossible. The ambiguity in the diffraction pattern itself can be avoided by choosing a wavelength short enough to produce at least seven lines on the pattern.
3.3 Crystal Structure of BaO:

The X-ray diffraction for BaO powder patterns are shown in Figure (3.1). These were obtained for BaO using an X-ray wavelength of ($\lambda=0.193609$ nm). After a few trials, the integers ($s$) listed in the third column of the Table (3.1), were found to produce the reasonably constant quotients listed in the fourth column, when divided into the observed ($sin^2\theta$) values. The fifth column lists the lattice parameter calculated from each line position, and the sixth column gives the Miller indices of each line.

The lattice parameter calculated from each the 11 peaks, shows the constant vales of (5.498 Å). It is clear from the miller indices, which is listed in sixth column that the crystal structure of BaO was Face Center Cubic. The sketch of this crystal structure is shown in Figure (3.2).

![Fig.(3.1): X-ray diffraction patterns for BaO powder.](image-url)
**Table (3.1):** Indexing of BaO (face center cube):

<table>
<thead>
<tr>
<th>Line no.</th>
<th>$\sin^2(\Theta)$</th>
<th>S</th>
<th>$\lambda^2/4a^2$</th>
<th>$a^{(\text{Å})}$</th>
<th>hkl</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.093</td>
<td>3</td>
<td>0.031</td>
<td>5.498</td>
<td>111</td>
</tr>
<tr>
<td>2</td>
<td>0.122</td>
<td>4</td>
<td>0.031</td>
<td>5.498</td>
<td>200</td>
</tr>
<tr>
<td>3</td>
<td>0.246</td>
<td>8</td>
<td>0.031</td>
<td>5.498</td>
<td>220</td>
</tr>
<tr>
<td>4</td>
<td>0.337</td>
<td>11</td>
<td>0.031</td>
<td>5.498</td>
<td>311</td>
</tr>
<tr>
<td>5</td>
<td>0.366</td>
<td>12</td>
<td>0.031</td>
<td>5.498</td>
<td>222</td>
</tr>
<tr>
<td>6</td>
<td>0.491</td>
<td>16</td>
<td>0.031</td>
<td>5.498</td>
<td>400</td>
</tr>
<tr>
<td>7</td>
<td>0.582</td>
<td>19</td>
<td>0.031</td>
<td>5.498</td>
<td>331</td>
</tr>
<tr>
<td>8</td>
<td>0.612</td>
<td>20</td>
<td>0.031</td>
<td>5.498</td>
<td>420</td>
</tr>
<tr>
<td>9</td>
<td>0.734</td>
<td>24</td>
<td>0.031</td>
<td>5.498</td>
<td>422</td>
</tr>
<tr>
<td>10</td>
<td>0.828</td>
<td>27</td>
<td>0.031</td>
<td>5.498</td>
<td>333,511</td>
</tr>
<tr>
<td>11</td>
<td>0.980</td>
<td>32</td>
<td>0.031</td>
<td>5.498</td>
<td>440</td>
</tr>
</tbody>
</table>

**Fig. (3.2):** Crystal structure of BaO sample.
3.4 Crystal Structure of NaO₂:

The X-ray diffraction for NaO₂ powder patterns are shown in Figure (3.3). The Figure shows that all lines which have mixed odd and even indices, such as 100, 110, etc., are absent from the pattern. Reference to the rules relating Bravais lattices to observed and absent reflections, given in Table (3.2), shows that the Bravais lattice of this specimen is Face Centered Cubic and the lattice parameter shows the constant values of (5.498Å). We now have certain information about the arrangement of atoms within the unit cell, and it should be noted that we have had to make use of observed line intensities in order to obtain this information. In this particular case, the observation consisted simply in noting which lines had zero intensity.

Fig.(3.3): X-ray diffraction patterns for NaO₂ powder.
Table (3.2): Indexing of NaO₂ (face center cube):

<table>
<thead>
<tr>
<th>Line no.</th>
<th>$\sin^2(\Theta)$</th>
<th>S</th>
<th>$\lambda^2/4a^2$</th>
<th>$a(^oA)$</th>
<th>hkl</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.093</td>
<td>3</td>
<td>0.031</td>
<td>5.498</td>
<td>111</td>
</tr>
<tr>
<td>2</td>
<td>0.122</td>
<td>4</td>
<td>0.031</td>
<td>5.498</td>
<td>200</td>
</tr>
<tr>
<td>3</td>
<td>0.246</td>
<td>8</td>
<td>0.031</td>
<td>5.498</td>
<td>220</td>
</tr>
<tr>
<td>4</td>
<td>0.337</td>
<td>11</td>
<td>0.031</td>
<td>5.498</td>
<td>311</td>
</tr>
<tr>
<td>5</td>
<td>0.366</td>
<td>12</td>
<td>0.031</td>
<td>5.498</td>
<td>222</td>
</tr>
<tr>
<td>6</td>
<td>0.491</td>
<td>16</td>
<td>0.031</td>
<td>5.498</td>
<td>400</td>
</tr>
<tr>
<td>7</td>
<td>0.586</td>
<td>19</td>
<td>0.031</td>
<td>5.498</td>
<td>331</td>
</tr>
<tr>
<td>8</td>
<td>0.616</td>
<td>20</td>
<td>0.031</td>
<td>5.498</td>
<td>420</td>
</tr>
<tr>
<td>9</td>
<td>0.738</td>
<td>24</td>
<td>0.031</td>
<td>5.498</td>
<td>422</td>
</tr>
<tr>
<td>10</td>
<td>0.834</td>
<td>27</td>
<td>0.031</td>
<td>5.498</td>
<td>333,511</td>
</tr>
<tr>
<td>11</td>
<td>0.986</td>
<td>32</td>
<td>0.031</td>
<td>5.498</td>
<td>440</td>
</tr>
</tbody>
</table>

3.5 Crystal Structure of MgS:

The X-ray diffraction for MgS powder patterns are shown in Figure (3.4). After a few trials, the integers (s) listed in the third column of the Table (3.3), were found to produce the reasonably constant quotients listed in the fourth column, when divided into the observed ($\sin^2\theta$) values. The fifth column lists the lattice parameter calculated from each line position, and the sixth column gives the Miller indices of each line.

The lattice parameter calculated form each the 10 peaks, shows the constant values of (5.25$^o$A). It is clear from the miller indices, which are listed in sixth column that the crystal structure of MgS was Face Center Cubic.
Fig. (3.4): X-ray diffraction patterns for MgS powder.

Table (3.3): Indexing of MgS (face center cube):

<table>
<thead>
<tr>
<th>Line no.</th>
<th>$\sin^2(\Theta)$</th>
<th>S</th>
<th>$\lambda^2/4a^2$</th>
<th>a (Å)</th>
<th>hkl</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.103</td>
<td>3</td>
<td>0.034</td>
<td>5.25</td>
<td>111</td>
</tr>
<tr>
<td>2</td>
<td>0.137</td>
<td>4</td>
<td>0.034</td>
<td>5.25</td>
<td>200</td>
</tr>
<tr>
<td>3</td>
<td>0.276</td>
<td>8</td>
<td>0.034</td>
<td>5.25</td>
<td>220</td>
</tr>
<tr>
<td>4</td>
<td>0.383</td>
<td>11</td>
<td>0.034</td>
<td>5.25</td>
<td>311</td>
</tr>
<tr>
<td>5</td>
<td>0.417</td>
<td>12</td>
<td>0.034</td>
<td>5.25</td>
<td>222</td>
</tr>
<tr>
<td>6</td>
<td>0.552</td>
<td>16</td>
<td>0.034</td>
<td>5.25</td>
<td>400</td>
</tr>
<tr>
<td>7</td>
<td>0.658</td>
<td>19</td>
<td>0.034</td>
<td>5.25</td>
<td>331</td>
</tr>
<tr>
<td>8</td>
<td>0.691</td>
<td>20</td>
<td>0.034</td>
<td>5.25</td>
<td>420</td>
</tr>
<tr>
<td>9</td>
<td>0.831</td>
<td>24</td>
<td>0.034</td>
<td>5.25</td>
<td>422</td>
</tr>
<tr>
<td>10</td>
<td>0.935</td>
<td>27</td>
<td>0.034</td>
<td>5.25</td>
<td>333,511</td>
</tr>
</tbody>
</table>
3.7 Crystal Structure of ZnZr:

The X-ray diffraction for ZnZr powder patterns are shown in Figure (3.5). Reference to the rules relating Bravais lattices to observed and absent reflections, given in Table (3.4), shows that the Bravais lattice of this specimen is Body Centered Cubic. The lattice parameter shows the constant values of \((5.498\,\text{Å})\). The sketch of this crystal structure is shown in Figure (3.6).

**Fig.(3.5):** X-ray diffraction patterns for ZnZr powder.

**Table(3.4):** Indexing of ZnZr (body center cube):

<table>
<thead>
<tr>
<th>Line no.</th>
<th>(\sin^2(\Theta))</th>
<th>S</th>
<th>(\lambda^2/4a^2)</th>
<th>(a(\text{Å}))</th>
<th>hkl</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.168</td>
<td>2</td>
<td>0.084</td>
<td>3.34</td>
<td>110</td>
</tr>
<tr>
<td>2</td>
<td>0.337</td>
<td>4</td>
<td>0.084</td>
<td>3.34</td>
<td>200</td>
</tr>
<tr>
<td>3</td>
<td>0.504</td>
<td>6</td>
<td>0.084</td>
<td>3.34</td>
<td>211</td>
</tr>
<tr>
<td>4</td>
<td>0.671</td>
<td>8</td>
<td>0.084</td>
<td>3.34</td>
<td>220</td>
</tr>
<tr>
<td>5</td>
<td>0.840</td>
<td>10</td>
<td>0.084</td>
<td>3.34</td>
<td>310</td>
</tr>
</tbody>
</table>
3.6 Crystal Structure of AgCd:

The X-ray diffraction for AgCd powder patterns are shown in Figure (3.7). After a few trials, the integers \((s)\) listed in the third column of the Table (3.5), were found to produce the reasonably constant quotients listed in the fourth column, to produce a constant lattice parameter from each line position, and then the Miller indices of each line can be calculated.

The lattice parameter calculated from all peaks, shows the constant values of \((3.34\text{oA})\). It is clear from the miller indices, which is listed in sixth column that the crystal structure of AgCd was Body Center Cubic.
Fig.(3.7): X-ray diffraction patterns for AgCd powder.

Table(3.5): Indexing of AgCd (body center cube):

<table>
<thead>
<tr>
<th>Line no.</th>
<th>$\sin^2(\Theta)$</th>
<th>S</th>
<th>$\lambda^2/4a^2$</th>
<th>$a(\text{oA})$</th>
<th>hkl</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.168</td>
<td>2</td>
<td>0.084</td>
<td>3.34</td>
<td>110</td>
</tr>
<tr>
<td>2</td>
<td>0.337</td>
<td>4</td>
<td>0.084</td>
<td>3.34</td>
<td>200</td>
</tr>
<tr>
<td>3</td>
<td>0.508</td>
<td>6</td>
<td>0.084</td>
<td>3.34</td>
<td>211</td>
</tr>
<tr>
<td>4</td>
<td>0.675</td>
<td>8</td>
<td>0.084</td>
<td>3.34</td>
<td>220</td>
</tr>
<tr>
<td>5</td>
<td>0.844</td>
<td>10</td>
<td>0.084</td>
<td>3.34</td>
<td>310</td>
</tr>
</tbody>
</table>

3.8 Crystal Structure of CuSc:

The X-ray diffraction for CuSc powder patterns are shown in Figure (3.8). After a few trials, the integers (s), lattice parameter, and the Miller indices of each line are calculated and listed in the Table (3.6). The lattice parameter calculated form all peaks, shows the constant values of (3.24\text{oA}).
From the miller indices, which is listed in sixth column the crystal structure of CuSc can be estimated to be Body Center Cubic.

**Fig.(3.8):** X-ray diffraction patterns for CuSc powder.

**Table(3.6): Indexing of CuSc (body center cube):**

<table>
<thead>
<tr>
<th>Line no.</th>
<th>Sin²(Θ)</th>
<th>S</th>
<th>λ²/4a²</th>
<th>a(Å)</th>
<th>hkl</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.178</td>
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<td>0.089</td>
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<td>110</td>
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<tr>
<td>2</td>
<td>0.357</td>
<td>4</td>
<td>0.089</td>
<td>3.24</td>
<td>200</td>
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<tr>
<td>3</td>
<td>0.534</td>
<td>6</td>
<td>0.089</td>
<td>3.24</td>
<td>211</td>
</tr>
<tr>
<td>4</td>
<td>0.715</td>
<td>8</td>
<td>0.089</td>
<td>3.24</td>
<td>220</td>
</tr>
<tr>
<td>5</td>
<td>0.894</td>
<td>10</td>
<td>0.089</td>
<td>3.24</td>
<td>310</td>
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</tbody>
</table>

**3.9 Crystal Structure of LaTe:**

The X-ray diffraction for LaTe powder patterns are shown in Figure (3.9). One possible structures were examined cubic. For each the 9-peaks, the lattice parameter of this sample which is (3.22 Å) was calculated. From the results obtained in Table (3.7), we can conclude that the crystal structure of LaTe is Simple Cubic. The sketch of this crystal structure is shown in Figure (3.10).
Fig.(3.9): X-ray diffraction patterns for LaTe powder.

Table(3.7): Indexing of LaTe (simple cube):

<table>
<thead>
<tr>
<th>Line no.</th>
<th>Sin²(Θ)</th>
<th>S</th>
<th>λ²/4a²</th>
<th>a(Å)</th>
<th>hkl</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
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<td>1</td>
<td>0.09</td>
<td>3.22</td>
<td>100</td>
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<tr>
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<td>2</td>
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<td>3.22</td>
<td>110</td>
</tr>
<tr>
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<td>0.273</td>
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<td>3.22</td>
<td>111</td>
</tr>
<tr>
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<td>3.22</td>
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<td>3.22</td>
<td>210</td>
</tr>
<tr>
<td>6</td>
<td>0.639</td>
<td>6</td>
<td>0.09</td>
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<td>211</td>
</tr>
<tr>
<td>7</td>
<td>0.723</td>
<td>8</td>
<td>0.09</td>
<td>3.22</td>
<td>220</td>
</tr>
<tr>
<td>8</td>
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<td>9</td>
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</tr>
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<td>0.904</td>
<td>10</td>
<td>0.09</td>
<td>3.22</td>
<td>310</td>
</tr>
</tbody>
</table>
3.10 Crystal Structure of ErTe:

The X-ray diffraction for ErTe powder patterns are shown in Figure (3.11). Examining the Simple Cubic structure, the integers \((s)\), lattice parameter, and Miller indices of each 8 line are calculated and listed in the Table (3.8). The lattice parameter shows the constant values of \((3.01\,\text{oA})\), indicating that the crystal structure of ErTe was Simple Cubic.

Fig.(3.10): Crystal structure of LaTe sample.

Fig.(3.11): X-ray diffraction patterns for ErTe powder.
Table (3.8): Indexing of ErTe (simple cube):

<table>
<thead>
<tr>
<th>Line no.</th>
<th>Sin²(Θ)</th>
<th>S</th>
<th>λ²/4a²</th>
<th>a(Å)</th>
<th>hkl</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
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<td>1</td>
<td>0.103</td>
<td>3.01</td>
<td>100</td>
</tr>
<tr>
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</tr>
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<td>0.308</td>
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<tr>
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<td>211</td>
</tr>
<tr>
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<td>0.103</td>
<td>3.01</td>
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</tr>
<tr>
<td>8</td>
<td>0.935</td>
<td>9</td>
<td>0.103</td>
<td>3.01</td>
<td>300</td>
</tr>
</tbody>
</table>

3-11 Conclusions:

Structures are most often determined by diffraction of X-rays from single crystals of the sample, although structure determination from polycrystalline powders is also important. The diffraction of this ray by crystal provide information about the internal structure of the crystal. The X-ray can be used, because there wavelength is so short that is the same order of magnitude as a lattice constant of crystal.

Powder patterns of cubic substances can usually be distinguished at a glance from those of noncubic substances, since the latter patterns normally contain many more lines. In addition, the Bravais lattice can usually be identified by inspection: there is an almost regular sequence of lines in simple cubic and body-centered cubic patterns, but the former contains almost twice as many lines, while a face-centered cubic pattern is characterized by a pair of lines, followed by a single line, followed by a pair, another single line, etc.

The crystal structure of some different binary system have been successfully determined from X-ray powder diffraction data. The analysis of the spectrums
shows that the (BaO, NaO₂, MgS) have Face Centered Cubic structures with the lattice parameters of (5.498, 5.498, 5.25) Å respectively, while the samples of (ZnZr, AgCd, CuSc) have Body Centered Cubic crystal structures with the values of lattice parameters (3.34, 3.34, 3.24) Å respectively, finally the samples of (LaTe, ErTe) have Simple Cubic with the lattice parameters (3.22, 3.01) Å respectively.
References

Books

1- Uri Shmueli, “Theories and Techniques of Crystal Structure Determination”, The moral rights of the authors have been asserted Database right Oxford University Press, (2007).


Internet

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2- http://www2.egr.uh.edu/~smotamar/XRay/XRay.html
3- http://www.answers.com/topic/crystal-structure
4- http://reference.iucr.org/dictionary/Bragg%27s_law
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