

# X-Ray Diffraction: A Comprehensive Explanation for Multipurpose Research

Adree Khondker<sup>1</sup>, Shakir Lakhani<sup>2</sup>

<sup>1</sup>Westdale S.S., Hamilton, ON, Canada

<sup>2</sup>Upper Canada College, Toronto, ON, Canada

**Abstract:** The explanation of X-Ray Diffraction in secondary schools is very standalone and becomes hard to apply to the context of experimental research. A simple and comprehensive explanation offers methods for educators to use in teaching secondary students about the contexts of X-Ray diffraction analysis. By offering derivations of the key formulas (i.e. Bragg's Law) a step-by-step guide is provided to usher students into X-Ray Diffraction research from both analytical and societal perspectives. The discrepancy of mathematics allows students to truly dive into the applications and process of utilization of X-Rays, without being overwhelmed by the complexity. Looking at both hard condensed matter and soft matter, a large overview is given on the type of research that can be completed with X-Ray diffraction such as micro technology, forensic sciences, and structural analysis. Analyzing sodium chloride through diffraction analysis gives educators gradual examples to use for teaching. Scattering data is also touched upon, to overall give both the context and concept of X-Ray Diffraction research, which is quintessential to our understanding in many variant fields of science.

**Keywords:** Diffraction, X-Ray, Bragg's Law, Lattice, Crystallography, and Constructive/Destructive Interference.

## I. INTRODUCTION

X-Rays were discovered by Wilhelm Rontgen in 1896 but it was not until 1913 when W.L. Bragg demonstrated the use of this newly discovered electromagnetic radiation for structural analysis. The most commonly asked question is how can a beam of X-Rays determine the structure of nanoscale objects. The common explanation is often misconceived by the general public due to the oversimplification. In this article, we will display a perspective on XRD that showcases the concept and context that describes the mechanisms at work in terms of structural analysis in an alternative way suitable for secondary school students. Through the step-by-step derivation of Bragg's Law, the fundamental equation behind XRD, a careful explanation of the analysis processes in place, along with an explanation of the differences between hard and soft matter, XRD can be explained correctly, and more importantly, easily. We believe that knowledge of the above information is key, as X-ray diffraction techniques and applications (later discussed in greater detail) have become, and are continuing to contribute heavily to modern-day science, and society in general.

## II. DERIVING AND EXPLAINING BRAGG'S LAW

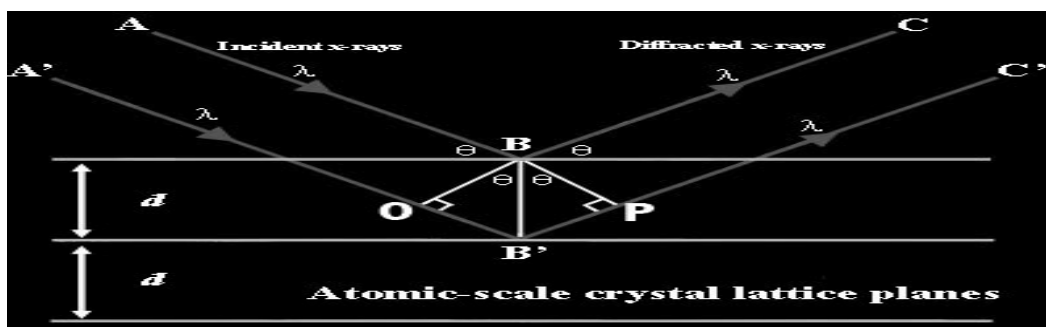


Fig. 1: Constructive Interference from in-phase reflected x-rays can be detected, and trigonometric equations are used to find distances between lattices.

Bragg's law refers to the equation  $n\lambda = 2d \sin\theta$ , derived by Bragg and his son to explain why the faces of crystals seem to reflect X-Ray beams at certain angles of incidence ( $\theta = 180x$  where  $x$  is an integer). The variable "d" refers to the distance between the atomic-scale crystal lattice planes shown above, and the variable "λ" refers to the wavelength of the X-ray beam incident on the surface of the crystal, where "n" is an integer.

Using reflection geometry and trigonometry from the image above, it is not difficult to derive Bragg's law. Incident rays A and A' are in phase and parallel up until A strikes the first plane at point B, while A' continues to the next plane and is scattered by the atom B'. If the two beams are to resume travelling parallel and in phase as C and C', the second beam must travel the extra distance marked on either side of B' by the right angle (let us call these points O and P) to point B', both while incident and after being scattered. If the phases of the two beams are to remain the same upon exit, then this extra distance must be a multiple (n) of λ.

Therefore,  $n\lambda = OB' + PB'$  (1)

To find OB' and PB', we can apply basic trigonometry. Noting that "d" is the hypotenuse of the right triangles BOB' and BPB', we can say that

$OB' = d \sin\theta$  (2)

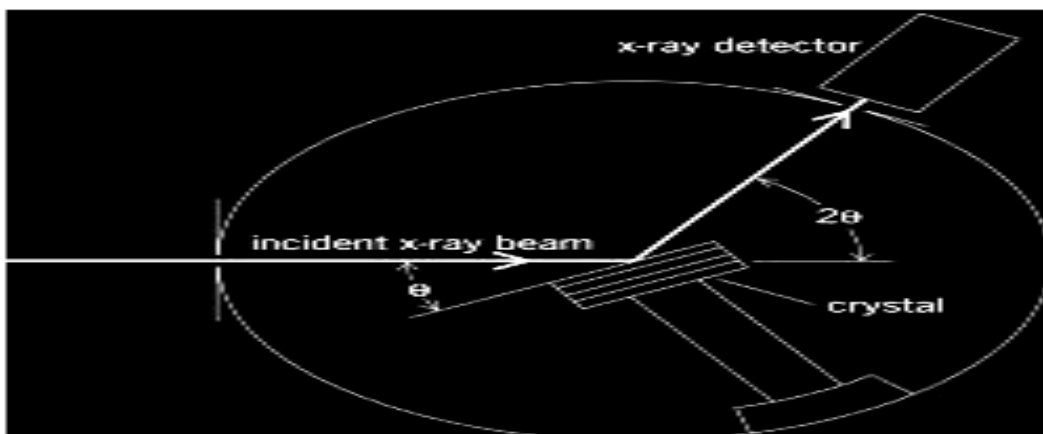
Since  $OB' = PB'$ , we can therefore state

$n\lambda = 2 OB'$  (3)

$= 2d \sin\theta$  [sub (2) into (3)]

As such, we have the derivation of Bragg's law.

It is important to note that in real life situations, when examining XRD patterns, very sharp peaks are surrounded by areas of high destructive interference, due to the many interfering atomic planes. In most theoretical models, only 2 planes of interference are shown, which is why the interference transition from constructive to destructive shown on graphs would be gradual as θ changes.



**Fig. 2:** The internals of XRD machinery that shows rotating arm from incident beam with corresponding detector via: x-ray diffraction. (n.d.) McGraw-Hill Dictionary of Scientific & Technical Terms, 6E. (2003). Retrieved February 3 2015

### III. OTHER APPLICATIONS OF X-RAY DIFFRACTION

XRD applications are becoming increasingly popular in today's scientific community, particularly within the contexts of pharmaceuticals, forensic science, geology, and microelectronics. It is increasingly vital for a secondary student to understand the context and uses of XRD.

In the pharmaceutical industry, XRD can be used to characterize the composition of a pharmaceutical due to the crystal structures present in it (the pharmaceutical). This characterization is also instrumental to the patenting of different active drugs, as the unique powder diffraction pattern is used to analyse the crystal structure. For compounds that consist of multiple components, the finite percentages of any active ingredients (along with packing ingredients) can also be accurately analysed in situ.

Furthermore, XRD has seen increased use in trace analysis in forensic science. Examples of its applications would be the analysis of paint flakes, hair, glass fragments, stains, loose powdered materials, etc. The analysis of these materials can play a large role in the evidence supporting the conviction of a person suspected of being involved in a crime.

On the opposite end of the spectrum, XRD has revolutionized the geological sciences with the utility of X-ray crystallography. XRD's ability to unambiguously characterize individual crystal structures is integral to any mineralogical study. Each mineral type has a unique XRD pattern, given by its characteristic crystal structure, allowing researchers to rapidly determine the proportion and types of minerals present within a sample of rock, soil, etc.

Finally, we note that the microelectronics industry uses single crystal substrates (gallium arsenide and silicon) in the production of integrated circuits. These materials need to be fully characterized by XRD topography in order to detect and image defects in a crystal, making XRD a reliable, and more importantly, non-destructive tool for characterizing important crystal specimens.

#### IV. INTERPRETING RESULTS WITH CRYSTALLINE LATTICES

Interpreting XRD can seem very overwhelming to a student, as the produced reflectivities show little apparent pattern in Figure X. However, the number and positions of peaks can show: crystal class, lattice type, and unit cell parameters, and the intensity of the peaks can show the types of atoms and the position of the atoms. It is important to define each of these terms for high school students to paint a complete picture of the process.

The positions and numbers of the peak give insight into the crystal family, which is a system to organize and classify the crystallinity of substances. Crystals are defined as substances with repeating structure, therefore a crystal can be divided into its repeating parts called motifs. From this, we attain a crystal class, there are 32 crystal classes, which in essence describe the symmetry of the motif if it was to be standalone. The lattice type is simply the shape the motif can be divided by, simply. For example, if the lattice type is hexagonal, if the motif were to be contained in a hexagon, the hexagon could be translated by integer amount to produce the structure of the crystal. It is important to note for students that this hexagon refers to a hexagonal prism, and that compounds are in three dimensions. Finally, the unit cell is the least volume consuming repeating structure of any solid, again consider a hexagonal unit cell.

The intensities of the peaks are determined by the atoms present and positions of the atoms. This is analogous to electrophoresis almost, where different sizes of particles are pulled to a specific distance due to their "uniqueness," likewise every atom or compound has a distinct size with a specific number of bonds which corresponds to the intensity of diffracted x-rays. Students typically have a hard time differentiating between bond energies and polarity-based diffraction. For students, it is easier to simply imagine energy being deflected from electron clouds, and in this sense, imagine x-rays are particles instead of waves. The more electrons they collide with result in higher number of diffracted beams, and this is most prominent in areas of high electron density. In other words, the most polar regions are shown by the increased intensity of diffracted electrons. Bond energies are a bit more counter-intuitive in terms of secondary school explanations. Intermolecular bonding such as hydrogen bonding, dipole-dipole bonding, and Van der Waals forces are found from specific intensities, it is analogous to a fingerprint. The positions of the atoms are used to determine the types of intermolecular bondings and the positions of covalent or ionic bonds.

To demonstrate this, we will analyze the reflectivity from the diffraction analysis of Sodium Chloride (NaCl). NaCl forms a simple lattice as seen in Figure 3a, that has 90° bond angles, and the reflectivity it produces is shown in 3b. Before we continue, it is important to mention that the following samples correspond to powder diffraction, which means x-rays that travel through the solid crystalline powder are being diffracted.

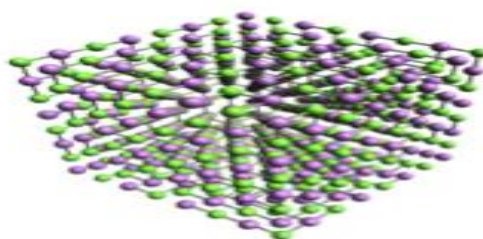


Fig. 3a: the crystal lattice of NaCl on Avogadro

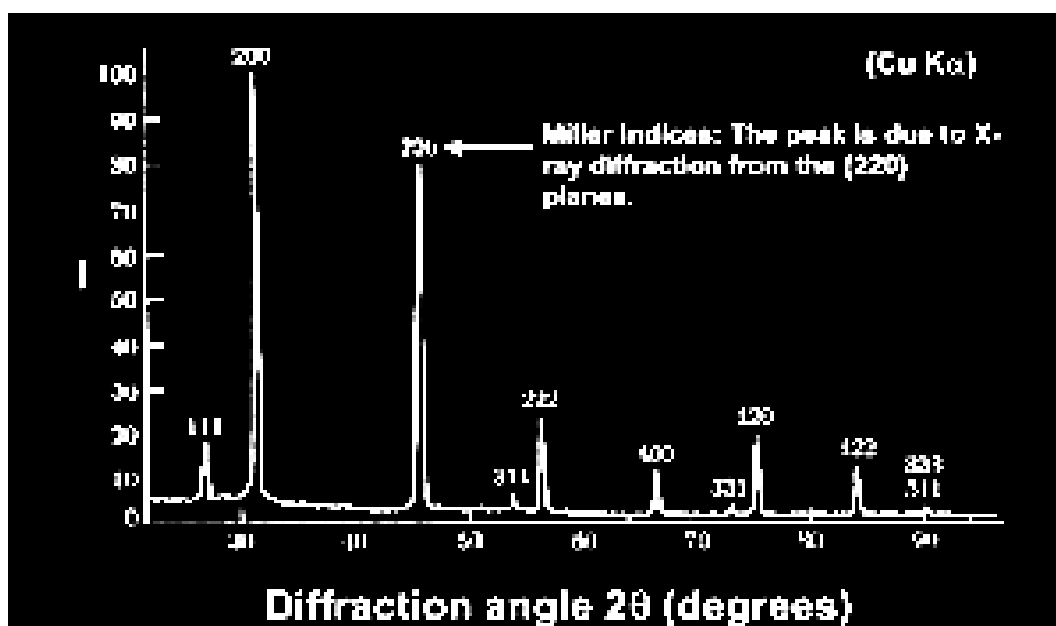


Fig. 3b: Scattering results from the diffraction of NaCl via: <http://web.pdx.edu/~pmoock/phy381/Topic5a-XRD.pdf>

To not overwhelm students, the essence should be extracted from the images. The Miller indices are the peaks of within the reflectivity's. Notice the angle  $2\theta$  as the x-axis, which represents double the angle of inclination which is set by the rotating arm in the diffractor. The critical thing for a student to attain from the above is that the rotating arm moves at an angle "up and down" while the detector moves at a corresponding angle where the highest intensity is shown by the peaks.

## V. HARD CONDENSED MATTER VERSUS SOFT CONDENSED MATTER

It is important to note the difference between hard matter and soft matter due to the dramatic difference in their respective reflectivities. Generally, hard matter allows for higher resolution due its more ordered arrangements. Secondary students are often familiar with copper as a metal - the reflectivity similar to Fig. 3a. On the other hand, soft matter is very disordered due to its fluid nature and must be prepared for XRD. This leads to lower resolution.

The resolution is distinct due to the broader peaks observed - in the reflectivity of Copper, sharp and distinct peaks are seen. The broader peaks are due to the nature of soft matter, for example, for the scanning of lipid membranes, they must be prepared to form lamellar and ordered stacks with wide-angle diffraction yet the resolution does not compare to powder diffraction of hard matter. Students fall under the trap to believe that resolution is equivalent to accuracy; however, the uncertainty is dependent on the sample preparation and the detector and not the type of matter.

The NaCl is an example of hard-condensed matter, due to the fact it is in an ordered lattice of alternating sodium and chlorine atoms which are charge centres which are shown by the 2D intensity profile.

## VI. CONCLUSION

XRD can be utilized throughout science in a variety of fields from forensic science to structural analysis, shedding light on the unseen at the Angstrom scale. Bragg's law is a powerful tool in understanding XRD, and allows the student to dive into the measurements that can be made by the method. The reflectivity resolution can be variant based on the type of matter, either hard condensed matter or soft matter, permitting different types of research from metals to membranous substances. It can be observed from sodium chloride the sharp peaks and Miller indices that determine the unit cell, crystal family, position of atoms, etc. For interested students, important future topics can be promoted as an in-class project such as: geometry differences in unit cells, Miller indices, XRD machinery, the composition of X-Rays, specific structures analyzed by XRD not mentioned. An educator should promote the beauty of XRD as well as its applications alongside the essential concepts behind the mathematics.

#### REFERENCES

- [1] Bragg WH (1912). "On the direct or indirect nature of the ionization by X-rays". *Phil. Mag.* 23 (136): 647
- [2] Bragg WL (1912). "The Specular Reflexion of X-rays". *Nature* 90 (2250): 410.
- [3] Crowfoot Hodgkin D (1935). "X-ray Single Crystal Photographs of Insulin". *Nature* 135(3415): 591.
- [4] Hull AW (1917). "The Crystal Structure of Iron". *Phys. Rev.* 9: 84.
- [5] Taylor G (2003). "The phase problem". *Acta Crystallographica D* 59 (11): 1881.
- [6] Patterson AL (1935). "A Direct Method for the Determination of the Components of Interatomic Distances in Crystals". *Zeitschrift für Kristallographie* 90: 517.
- [7] Charles W. Carter and Robert M. Sweet., ed. (1997). *Macromolecular Crystallography, Part A (Methods in Enzymology, v. 276)*. San Diego: Academic Press.
- [8] Charles W. Carter Jr., Robert M. Sweet., ed. (1997). *Macromolecular Crystallography, Part B (Methods in Enzymology, v. 277)*. San Diego: Academic Press.
- [9] B.E. Warren (1969). *X-ray Diffraction*. New York.
- [10] Zachariasen WH (1945). *Theory of X-ray Diffraction in Crystals*. New York: Dover Publications.
- [11] Seiler P, Dunitz JD (1979). "A new interpretation of the disordered crystal structure of ferrocene". *Acta Crystallographica B* 35 (5): 1068.