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A PATH FOR HORIZING YOUR INNOVATIVE WORK

KNOWLEDGE WORLD OF CRYSTALS ---- ATOMS IN ORDER

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Abstract: The year 2014 has been declared as the 'International Year of Crystallography' by the United Nations to celebrate the completion of 100 years of the discoveries that led to the emergence and development of the knowledge domain of crystallography. In this paper we review how these groundbreaking discoveries motivated pure and applied research opening new frontiers of knowledge that led to rapid developments in science and technology. The review discusses the chronology of the pioneering works of several scientists that shaped our present knowledge world of crystals. An open knowledge spiral is envisaged that embodies this world and can motivate free and flexible learning of crystals by interested learners.

Keywords: x-rays, crystallography, diffraction, x-ray spectroscopy, x-ray spectrometry, protein crystallography, crystal engineering and crystal knowledge spiral



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INTRODUCTION

The declaration of year 2014 as the International Year of Crystallography by the United Nations has attracted the attention of people in general and scientific community in particular towards the knowledge domain of crystallography.^[1,2] Crystals had been a subject of curiosity for humans since antiquity due to their beautiful shapes and symmetries. The underlying cause of this beauty has been attributed to the orderly arrangements of atoms in crystals. These arrangements always lead to the lowest energy and hence the greatest stability. Crystalline state is thus the most favoured state of solids in nature. More than 90% of the naturally occurring solids such as minerals, rocks, sand, ice, clay, gems, metals, carbon and salts are crystalline. Crystals are also produced by some living organisms, e.g., calcite produced by most molluscs.

Our understanding of the crystalline structures has grown rapidly after the groundbreaking discoveries by pioneers like Laue and Bragg about hundred years back. On one hand these discoveries paved way for a lot of research into the invisible structural world of the myriad crystalline matter around us and on the other hand the knowledge output of this research found wide applications in various fields of technology. In this paper we review how our basic knowledge of crystals brought forth through pure research opened new frontiers of applied research and contributed to several new developments in science and technology.

BASIC IDEAS

The orderly arrangement of atoms in crystals is studied through the basic concepts of *lattice*, *basis* and *unit cell*. A lattice is an infinite array of points in space in which each point has surrounding exactly identical to the surrounding of any other point in that array. Basis means either an atom or a group of atoms, which when attached to each lattice point generates a crystal. Unit cell is the smallest building block of a crystal so formed. In 1845, Auguste Bravais, a French physicist, showed that there can be only 14 such unique lattices.^[3] All crystalline materials (excluding quasicrystals) fit in one of these arrangements.

Bravais lattices can be categorized into seven 'crystal systems', depending on the relations between distances between points in the unit cell, i.e., edges of the unit cell and between the angles between these edges - called the *lattice parameters*. *Cube* is the simplest and the most symmetric system for which all the edges of the unit cell are equal and are mutually perpendicular. *Triclinic* system is the least symmetric and most complicated as all the edges of the unit cell are unequal and also the angles between them are unequal.

Diffraction is another key concept in the study of crystals. The phenomenon of diffraction has been well studied for light since it was first discovered by the Italian physicist, Francesco Grimaldi, in 1665.^[4] Diffraction means bending and it was found that any wave can diffract if its wavelength is of the order of the size of the obstacle which it encounters (see Table 1). The pattern produced by interference of the secondary waves produced by the obstacle is called the *diffraction pattern*.

Wave	Order of wavelength	Examples of obstacles that can diffract
Sound wave	few meters	any obstacle with size of the order of meters
Light wave	4000 A [°] to 7000 A [°]	thin slits, sharp edges, <i>gratings</i> (large number of lines ruled on a small glass plate so that the separation between adjacent lines is of the order of wavelength of light)

Table 1 Examples of obstacles causing diffraction of different waves

When x-rays were discovered by the German physicist, W. C. Röntgen, in 1895^[5], there was an excitement about knowing the conspicuous properties of this new type of radiation. Questions like what is the wavelength range of x-rays and whether x-rays can be diffracted aroused the curiosity of researchers. We see next how these questions led to the groundbreaking discoveries by Laue and Bragg.

GROUNDBREAKING DISCOVERIES BY LAUE AND BRAGG

Max von Laue, a German physicist, who had used gratings extensively to study the diffraction of light, was one of the earliest to probe into these questions.^[6] Laue knew from his experimental work in Roentgen's laboratory that the wavelength of x-rays was of the order of few angstroms. In 1912, while discussing with Paul Ewald, a German born US physicist, about the latter's doctoral thesis, Laue was informed that the distance between the atoms in a crystal lattice was also of the same order. Laue got a brilliant idea: why not use crystal as a natural grating to diffract x-rays?

Motivated by this idea Laue performed an experiment in which he exposed a copper sulphate crystal to x-rays and recorded its diffraction pattern. The pattern showed a large number of systematically arranged spots. Laue used his knowledge of the diffraction of light by a grating and accounted for these spots on the basis of the phenomenon of diffraction of x-rays by a

crystal. Laue's contribution marks the birth of *x-ray crystallography*. He was awarded the Nobel Prize in Physics in 1914 for this pioneering contribution.

Max von Laue

William Henry Bragg

Laue's discovery generated a lot of enthusiasm about further research in x-ray crystallography. The father and son team of William Henry Bragg and William Lawrence Bragg took a pioneering interest in this research.^[7] Henry Bragg developed a *spectrometer* (Fig. 1) in which x-rays were incident on the crystal to be probed and the intensities of diffracted beam were measured as a function of the angle through which the x-ray beam got diffracted. When x-rays were incident on crystals at specific angles, intense peaks of diffracted radiation were observed at certain specific wavelengths.

Fig. 1 : Bragg's X-ray Spectrometer

Lawrence Bragg provided a successful explanation of these observations to the Cambridge Philosophical Society on 11^{th} November, 1912 using a law derived by him for this purpose, later known as *Bragg's law*: $2d \sin\theta = n\lambda$. In the model proposed by Bragg a crystal is assumed as sets of parallel atomic planes that diffract x-rays at different angles. Bragg's law relates these angles, θ (called the *Bragg angle* or *angle of diffraction*) with the spacings between the planes responsible for diffraction, d; the wavelength of x-rays, λ ; and the order of diffraction, n (Fig. 2).









Considering that the impact of crystallography is present everywhere in our daily lives, in modern drug development, nanotechnology and biotechnology, and underpins the development of all new materials, from toothpaste to aeroplane components,

Considering also the significance of the scientific achievements of crystallography, as illustrated by twenty-three Nobel Prizes awarded in the area, and that crystallography is still

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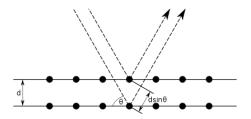


Fig. 2: Bragg's diffraction (Filled circles denote atoms and dashed lines represent monochromatic x-rays.)

The Braggs were awarded the Nobel Prize in physics in 1915 for their discovery. In the history of Nobel prizes, this is the only instance of a father-son team jointly receiving the honour. Secondly, Lawrence Bragg is the first Australian and the youngest Nobel laureate in science (25 years old then) so far. Considering the groundbreaking role of the discoveries of Laue and Braggs in the development of x-ray crystallography United Nations adopted a resolution on 12th July 2012 to declare 2014 as the International Year of Crystallography (see box 1).^[1]

Box 1 : Draft of the UN Resolution

in particular, in our knowledge of crystallography,

health, as well as solutions for plant and soil contamination,

Stressing that education about and the application of crystallography are critical in addressing challenges such as diseases and environmental problems, by providing protein and small molecule structures suited for drug design essential for medicine and public

Recognizing that humankind's understanding of the material nature of our world is grounded,

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fertile ground for new and promising fundamental research,

Considering further that 2014 marks the centenary of the beginning of modern crystallography and its identification as the most powerful tool for structure determination of matter,

Being aware that 2014 provides an opportunity to promote international collaboration as part of the sixty-fifth anniversary of the founding of the International Union of Crystallography,

Noting the broader welcome by the crystallographic community worldwide of the idea of 2014 being designated as the International Year of Crystallography,

Recognizing the leading role of the International Union of Crystallography, an adhering body of the International Council for Science, in coordinating and promoting crystallographic activities at the international, regional and national levels around the world,

1. Decides to proclaim 2014 the International Year of Crystallography;

2. -----

121st plenary meeting

3 July 2012

NEW FRONTIERS OF KNOWLEDGE

A closer look at Bragg's simple relation: $2d \sin\theta = n\lambda$ gives us an idea about the new routes it can open to further research. One route is to use a crystal of known structure, i.e., known d, measure θ and calculate λ . The cause of λ emitted by a material is the transition of electron from a higher energy level to a lower energy level in that material. Thus the knowledge of λ helps in working out the details of different electronic energy levels in different materials. This route led to the development of new domains of knowledge called x-ray spectroscopy and x-ray spectrometry. Braggs themselves pioneered the development in these domains by determining x-ray wavelengths of several elements precisely. A typical x-ray emission spectrum of any element is shown in Fig. 3. It consists of a continuous spectrum superimposed by the characteristic lines, labeled as K_{α} and K_{β} in the figure. The continuous x-rays find application in radiography whereas the characteristic ones are used in x-ray diffraction studies.

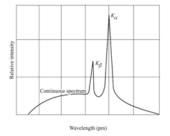
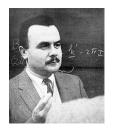


Fig. 3: A typical x-ray emission spectrum

When x-rays fall on any element they cause a secondary radiation, which is characteristic of that element. This phenomenon, known as *x-ray fluorescence*, is widely used for analysis in geochemistry, forensic science and archeology. In 1908, C. G. Barkla, a British physicist carried out a thorough investigation of the characteristic radiations in large number of substances.^[8] These researches deepened our understanding of the inner structure of atoms and opened a vast treasure of scientific knowledge. He was awarded the Nobel Prize in physics in 1917 for his contribution of far reaching significance.

The concept of wavelength is not limited to radiation alone. In 1924 a French physicist, Louis de Broglie postulated in his doctoral thesis that all matter has wave properties. This implied that particles (e.g., electrons) can be used as waves that can be diffracted by crystals. American physicists, Clinton Davisson and Lester Germer worked with this motivation during 1923 - 1927 and succeeded in diffracting electrons using nickel crystal. They determined the experimental value of wavelength of electrons using Bragg's relation and the theoretical value using the de Broglie hypothesis. Close agreement between the two values validated de Broglie hypothesis. De Broglie and Davisson *et. al.* received Nobel Prizes in physics in 1929 and 1937 respectively.^[9]

Discovery of *matter waves* opened up new vistas in the fields of microscopy and materials characterization. A microscope was developed in 1932 using the wave nature of electrons. Since then many developments have taken place such as *scanning electron microscopy* and *tunneling electron microscopy* that can facilitate high resolution imaging leading to significant advances in our knowledge of microworld. Various new techniques such as *electron diffraction* and *neutron diffraction* based on the wave character of particles were developed for material characterization. The 1994 Nobel Prize was awarded in Physics jointly to an American physicist, Clifford Shull and a Canadian physicist, Bertram Brockhouse for pioneering contributions to the development of *neutron diffraction* techniques for studies of condensed matter.^[10] In these techniques a beam of neutrons directed at a given material undergoes scattering by atoms in that material and a diffraction pattern of the atoms' positions can then be obtained.



Bertram Brockhouse



Clifford Shull (right) working with a double-crystal neutron spectrometer

Another route is to use x-rays of known λ , measure θ and calculate d. The knowledge of d (in fact, several d's as there are several atomic planes in a crystal) helps in working out the arrangement of individual atoms inside the crystals and thus in identifying their structures. This route led to the development of x-ray crystallography. X-ray crystallography opened the floodgates of knowledge of the world of crystalline materials. Out of the 500,000 structures that have been recorded in the Cambridge Structural Database, over 99% have been determined by x-ray diffraction. In the next section we review how the developments in the knowledge world of crystals influenced almost all branches of science and technology.

KNOWLEDGE WORLD OF CRYSTALS

The Braggs determined the first structure of a crystal known as *zincblende*. However, their most famous investigation was finding the structure of table salt in 1914, which they found to belong to the simple cubic lattice if the difference between the Na⁺ and Cl⁻ ion positions is ignored.^[11] In the same year, the Braggs also discovered the structure of diamond as the tetrahedral arrangement of carbon atoms held together by strong *covalent bonds* that were estimated to be 1.52 A° long. Two years later structure of graphite was revealed as atoms arranged in stacked sheets such that within the sheet, atoms are held together by strong *covalent bonds*. These revelations helped to understand why diamond is strong in all directions whereas graphite is soft and easy to cleave though both are pure carbon.

In 1916, Peter Debye, a Dutch-American physicist and his student and a Swiss physicist, Paul Scherrer, developed a new technique called the *powder diffraction method* in which the material could be taken in a powder form so that reflections of x-rays of known wavelength from different planes of atoms in the crystal could be simultaneously measured.^[12] This method was also developed independently by Albert Hull in the U.S. The Debye-Scherrer method was technically more useful as it allowed measurements of structural changes arising during various treatments of metals and alloys. It was thus widely used in mineralogy and chemistry for the

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identification of various minerals and chemical compounds. Debye received the Nobel Prize in Chemistry in 1936.



Peter Debye



Paul Scherrer

X-ray crystallography brought forth the atomic order of several solids in the following years. It was found that most metals crystallize in *face centered cubic* (eg., aluminum, copper, gold, iridium, lead, nickel, platinum, silver, etc.) and *hexagonal close packed* (e.g., beryllium, cadmium, magnesium, titanium, zinc, zirconium, etc.) structures due to the maximum packing of atoms (74 %) achievable with these structures.

After understanding simple crystal structures scientists were now keen to know about more complicated structures. The structures of silicates and garnets were discovered by 1924 benefiting the fields of mineralogy and metallurgy. The structure of hexamethylbenzene was determined by Dame Lonsdale, a British crystallographer, in 1928 that established the hexagonal symmetry of benzene.^[13] She was the first woman president of the International Union of Crystallography.



Dame Lonsdale

In 1926, James Sumner, an American chemist, demonstrated for the first time that an enzyme could be isolated and crystallized for which he shared the Nobel Prize in Chemistry with two American biochemists, John Northrop and Wendell Stanley, in 1946.^[14] He worked with an enzyme called *urease* and also provided the first experimental proof using chemical tests that an enzyme is a protein. Northrop repeated this for the gastric enzyme *pepsin* in 1929 and again for the first virus called *bacteriophage* in 1938.

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James Sumner John Northrop Wendell Stanley

Linus Pauling, an American chemist, determined structure of minerals using x-ray diffraction.^[15] He published seven papers in this area as a graduate student at Caltech and wrote a thesis, '*The determination with x-rays of the structures of crystals*' in 1925. In 1951, Pauling, along with Robert Corey and Herman Branson proposed the model of the *alpha helix* and *beta sheet* as primary structures in the protein secondary structure. Pauling received the Nobel Prize in Chemistry in 1954. He also received the Noble Prize for peace in 1962. He is the only person to date to be awarded two unshared Nobel Prizes.



Linus Pauling

Pauling was interested in studying protein structures to confirm his observation that the hemoglobin molecule changes structure when it gains or loses an oxygen atom. However, these structures were less amenable to this technique and it was John Kendrew, an English biochemist, who succeeded in determining the first atomic structure of protein (*myoglobin*) using x-ray crystallography.^[16] Kendrew shared the 1962 Nobel Prize for Chemistry with Max Perutz, a British molecular biologist. Their work was done at the Medical Research Council (MRC) Laboratory of Molecular Biology, created under the direction of Lawrence Bragg.



John Kendrew

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Perutz did his Ph. D. work under the guidance of William Bragg. Bragg also helped Perutz in getting financial support from the MRC to establish the Molecular Biology Unit at the Cavendish Laboratory to undertake research into the molecular structures of biological systems. This unit attracted several eminent researchers that included Francis Crick and James Watson. X-ray crystallography is now routinely used to determine the molecular structures of thousands of proteins every year. This research is useful in several diseases like sickle cell anemia and other neurodegenerative diseases.



Max Perutz

X-ray crystallography played an important role in one of the most revolutionary discoveries, the discovery of the structure of DNA, the chemical unit of life. In 1953, two English molecular biologists, Francis Crick and Maurice Wilkins and an American molecular biologist, James Watson reported the helical structure of DNA for which they were awarded the Nobel Prize for Physiology or Medicine in 1962.^[17]



Francis Crick Maurice Wilkins James Watson

Dorothy Hodgkin, a British Chemist, was one of the first persons to use x-ray crystallography for the study of structure of an organic compound.^[18] She developed a passion for chemistry from a young age and demonstrated the power of crystallographic techniques by deciphering the structures of *cholesterol* in 1937 and *penicillin* in 1946. She and her coworkers also determined the structure of vitamin B12 in 1956 after a thorough study that lasted for ten years. They also resolved the intricate structure of *insulin* in 1969 after a painstaking research of 35 years. Hodgkin was awarded the Nobel Prize in Chemistry in 1964 for her pioneering role in the development of *protein crystallography*.



Dorothy Hodgkin

X-ray crystallography started being widely used in biological research because the function of a biological molecule depends crucially on its structure. Christian Anfinsen, an American biochemist, worked with this motivation to explore the relationships between structure and function in proteins.^[19] In 1961 he showed that all the information required by protein to adopt its final conformation is encoded in its amino-acid sequence. He was awarded the 1972 Nobel Prize in Chemistry for his work on *Folding of protein chains*. In the 1950s, William Lipscomb, an American chemist determined the structure of *boranes* using x-ray crystallography and developed theories to explain their bonds for which he received the Nobel Prize in Chemistry in 1976.^[20] Linus Pauling was his Doctoral Advisor.



William Lipscomb Christian Anfinsen

The field was further advanced though the development of *crystallographic electron microscopy* in 1968 by Aaron Klug, a British chemist and biophysicist.^[21] This method uses a transmission electron microscope for imaging and is useful for studies of very small crystals because of the fact that electrons interact more strongly with atoms than x-rays. Klug was awarded the Nobel Prize in Chemistry for 1982 for *development of crystallographic electron microscopy and discovery of the structure of biologically important nucleic acid-protein complexes.* Nucleic acids are large biological macromolecules that encode, transmit and express genetic information in all forms of life and their knowledge constitutes the foundation for research in genome and forensic sciences, pharmaceutics and biotechnology.



Aaron Klug

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In 1985, Herbert Hauptman, an American mathematician and Jerome Karle, an American chemist jointly received the Nobel Prize in Chemistry for *development of direct methods for the determination of crystal structures*.^[22] These methods facilitate direct determination of crystal structures and are routinely used today to solve complicated structures. They have played a major role in the development of new pharmaceutical products and other synthesized materials.



Herbert Hauptman



Jerome Karle

Between 1982 and 1985, three German biochemists, Johann Deisenhofer, Hartmut Michel and Robert Huber, used x-ray crystallography to determine for the first time the arrangement of the more than 10,000 atoms that form the crystal structure of a protein complex found in certain photosynthetic bacteria.^[23] The knowledge of such a protein complex, called a *photosynthetic reaction centre*, is useful in understanding the mechanisms of photosynthetic processes of plants and bacteria. These three scientists were awarded the Nobel Prize in Chemistry in 1988.



Robert Huber

In 1991, Pierre-Gilles de Gennes, a French physicist was awarded the Nobel Prize in Physics for his discovery that methods developed for studying order phenomena in simple systems can be generalized to more complex forms of matter, in particular to liquid crystals and polymers.^[24]



P G de Gennes

As mentioned earlier, structures of diamond and graphite were already understood by x-ray crystallography as two allotropic forms of carbon. In 1985, Robert Curl, Richard Smalley (both professors at Rice University) and Harold Kroto (professor at the University of Sussex) reported the discovery of the third allotropic form of carbon, " C_{60} : Buckminsterfullerene".^[25] The molecule is named after Buckminster Fuller who created the famous geodesic dome structures, the architecture of which the molecule resembles. The trio was jointly awarded the Nobel Prize in Chemistry in 1996 for this discovery. These pioneering researches led to the development of the Rice Center for Nanoscience and Technology at Rice University.



Robert Curl Richard Smalley Harold Kroto

In 1998, Roderick MacKinnon, professor of Molecular Neurobiology and Biophysics at Rockefeller University, and his colleagues used x-ray crystallography to determine the structure of a *potassium channel*.^[26] MacKinnon earlier did his M.D. but got more interested in research than in the medical profession and started pursuing research in the interaction of the potassium channel with a specific toxin derived from scorpion venom. The knowledge of potassium channels is useful in the study of nervous system and heart. MacKinnon shared the Nobel Prize in Chemistry with Peter Agre, an American physician and molecular biologist, in 2003.

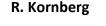


Roderick MacKinnon Peter Agre

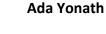
In 2006, Roger Kornberg, an American biochemist received the Nobel Prize in Chemistry for his studies of the process by which genetic information from DNA is copied to RNA, *"the molecular basis of eukaryotic transcription"*.^[27] Kornberg used x-ray crystallography to identify the role of



RNA polymerase II and other proteins in transcribing DNA thus bringing out how transcription works at a molecular level.



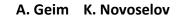
In 2009, the Nobel Prize in Chemistry was awarded to V. Ramakrishnan, an Indian-born American and Thomas Steitz, British structural biologist and Ada E. Yonath, an Israeli crystallographer for studies of the structure and function of the ribosome.^[28] Yonath became the first Israeli woman to win the Nobel Prize. In 1970, she established the protein crystallography laboratory in Israel. She introduced a novel technique called cryo bio-crystallography for enabling ribosomal crystallography.



Review Article

V. Ramakrishnan

The 2010 Nobel Prize in physics was awarded to Andre Geim and Konstantin Novoselov, of the University of Manchester, for groundbreaking experiments regarding the two-dimensional material graphene.^[29] Graphene is another crystalline allotrope of carbon in the form of about one atom thick, nearly transparent sheet. Due to its remarkable strength, very low weight, high electrical and thermal conductivity, it has many potential industrial applications, e.g., as bioelectric sensory devices, which find wide usage in LCD touch screens.











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The basics of crystallography were rewritten in 1982, when Dan Shechtman, an Israeli material scientist discovered new materials called the *quasicrystals*.^[30] He found that a rapidly solidified alloy of Al with 10-14 % Mn possesses an *icosahedral symmetry* in combination with long range order, which opened the new field of quasicrystalline materials. Linus Pauling used to oppose Shechtman's idea of quasi crystals vehemently and is even quoted as saying, "There is no such thing as quasicrystals, only quasi-scientists." However, Shechtman proved him wrong and was awarded the Nobel Prize in Chemistry in 2011 for his significant discovery. This discovery enthused scientists to search for the occurrence of quasicrystals in nature and their potential applications. Today quasicrystals are known to occur in nature and are used in a large number of applications that include the formation of steel used for fine instrumentation and non-stick insulation for electrical wires and cooking equipment.



Shechtman (left) explaining the structure of quasicrystals

The 2012 Nobel Prize for Chemistry was awarded to Robert Lefkowitz, an American physician and biochemist and Brian Kobilka, an American physiologist, for *studies of G-protein-coupled receptors (GPCRs)*.^[31] GPCRs are receptor proteins present on the cell surface that can sense molecules outside the cell and initiate a cellular response. Our understanding of the crystal structures of GPCRs and the mechanisms by which they work has helped immensely in pharmaceutical research and drug discovery. Today, more than 40 % of the drugs sold worldwide for various human disorders are molecules that target GPCRs. They are designed to "fit" like keys into the similarly structured locks of Lefkowitz' receptors.^[32]



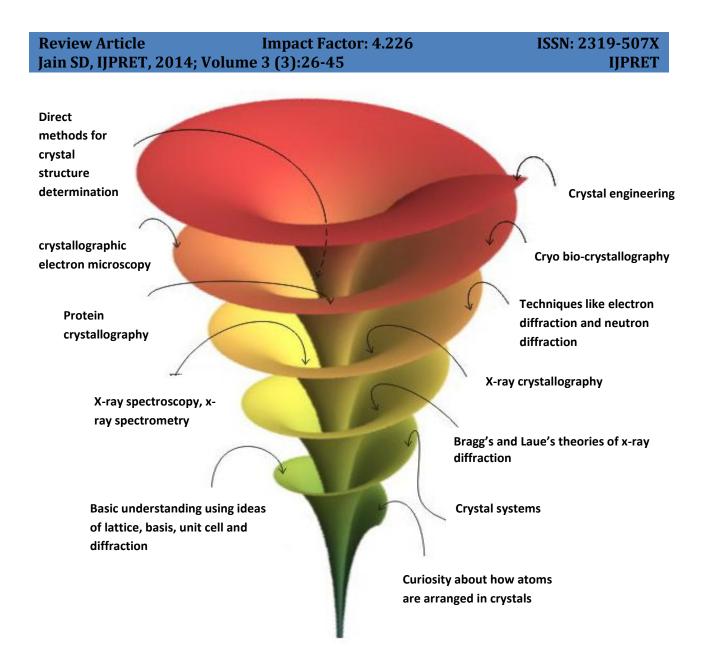
Robert Lefkowitz

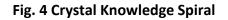
CRYSTALS AROUND US

The discussion so far convinces us about how our understanding of the crystalline matter has been enriched in the last hundred years influencing almost all walks of life. Today we find crystalline materials everywhere in use around us. Few examples are piezoelectric crystals used in radio transmitters and timepieces for producing ultrasonic waves, Nichol prisms used in polarimetry for polarizing light, crystalline semiconductors used in solar photovoltaic panels for converting sunlight into electricity and zeolites used in petroleum refinement to obtain better and cleaner fuel.

X-ray crystallography has also been used recently to study the mineral composition of planets. On 17th October, 2012, the Curiosity rover on the planet Mars performed the first x-ray diffraction analysis of Martian soil, which revealed the presence of minerals such as feldspar, pyroxenes and olivine. The design and synthesis of molecular solid-state structures with desired properties based on an understanding and exploitation of intermolecular interactions is known today as *Crystal engineering*.

As our knowledge world of crystals gets enriched an open *crystal knowledge spiral* can be envisaged (Fig, 4) that embodies this world and can motivate free and flexible learning of crystals by interested learners. Such a spiral can be implemented in a *crystal knowledge centre* as has been suggested in our recent work about other knowledge domains.^[33] Such a center envisages converting the hobbies and interests of young researchers in growing crystals and peeping into what they look like (i.e., their structures) into professions based on crystallography.





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