



Sudan University of Science and Technology



College of Graduate Studied

Studying the Crystalline Structure of Materials Using X - ray Diffraction

دراسة التركيب البلوري للمواد باستخدام حيود الاشعه السينية

Submitted in partial fulfillment of the requirement for the degree of
master by

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June 2017

بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

الايه

قال تعالى:

﴿ اللهُ نُورُ السَّمَوَاتِ وَالْأَرْضِ مِثْلُ نُورِهِ كَمِشْكُوتٍ فِيهَا مِصْبَاحٌ
الْمِصْبَاحُ فِي زُجَاجَةٍ الزُّجَاجَةُ كَأَنَّهَا كَوْكَبٌ دُرِّيٌّ يُوقَدُ مِنْ شَجَرَةٍ مُبْرَكَةٍ
زَيْتُونَةٍ لَا شَرْقِيَّةٍ وَلَا غَرْبِيَّةٍ يَكَادُ زَيْتُهَا يُضِيءُ وَلَوْ لَمْ تَمْسَسْهُ نَارٌ نُورٌ
عَلَى نُورٍ يَهْدِي اللهُ لِنُورِهِ مَنْ يَشَاءُ وَيَضْرِبُ اللهُ الْأَمْثَلَ لِلنَّاسِ وَاللَّهُ

بِكُلِّ شَيْءٍ عَلِيمٌ ﴿٣٥﴾

صدق الله العظيم

[سورة النور]

Acknowledgements

I would like to acknowledge the following people for their valuable contribution towards the completion of my Master work.

- *My academic promoter, Dr. Amel Abdallah for her tireless guidance, discussions and support throughout this study.*
- *My teacher Muna to help me to conduct experiments.*
- *My parents and my husband for their understanding, never ending love and encouragement throughout my studies and beyond.*

Abstract

The objective of this work is to study the crystalline structure of the materials as well as their properties and the components of the material, three different samples (ceramic, mica, chromium) were used. The properties and components of each sample were studied and the crystalline structure, using X-ray diffraction and Bragg law was investigated.

From Bragg law, the interplanar spacing (d) calculated, it was found that (d) for the first sample equal 1.3698nm, which is the same for the standard data that stands for Mica, while (d) for the second sample is equal to 0.9096nm which is identical to the ceramics according to standard data. The interplanar spacing (d) for the third samples showed the Chromium element

المستخلص

تم دراسة دراسة التركيب البلوري للمواد كما درس خصائصها و العناصر المكونه لها، باستخدام ثلاثه عينات مختلفه، تم التعرف علي خصائص و مكونات كل عينه و دراسة التركيب البلوري لها و ذلك باستخدام ظاهرة حيود الاشعه السينيه و قانون براغ.

ومن قانون براغ تم حساب المسافه البينية للذرات (d) و وجد ان (d) للعينه الاولي تساوي (nm) 1,3698 و هي مطابقة لعنصر المايكا وفقا للبيانات العالميه. بينما للعينه الثانيه وجد انها تساوي (nm) 0,9096 و هي مطابقة للسيراميك بينما العينه الثالثه و جد انها مطابقة لعنصر الكروم.

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Chapter One

Introduction

1.1 History of X-ray

German physicist Wilhelm Röntgen is usually credited as the discoverer of X-rays in 1895, because he was the first to systematically study them, though he is not the first to have observed their effects. He is also the one who gave them the name "X-rays" (signifying an unknown quantity) though many others referred to these as "Röntgen rays" (and the associated X-ray radiograms as, "Röntgenograms") for several decades after their discovery and even to this day in some languages, including Röntgen's native German.

X-rays were found emanating from Crookes tubes, experimental discharge tubes invented around 1875, by scientists investigating the cathode rays, that is energetic electron beams, that were first created in the tubes. Crookes tubes created free electrons by ionization of the residual air in the tube by a high DC voltage of anywhere between a few kilovolts and 100 kV. This voltage accelerated the electrons coming from the cathode to a high enough velocity that they created X-rays when they struck the anode or the glass wall of the tube. Many of the early Crookes tubes undoubtedly radiated X-rays, because early researchers noticed effects that were attributable to them, as detailed below. Wilhelm Röntgen was the first to systematically study them, in 1895.

The discovery of X-rays stimulated a veritable sensation. Röntgen's biographer Otto Glasser estimated that, in 1896 alone, as many as 49 essays and 1044 articles about the new rays were published. This was probably a conservative estimate, if one considers that nearly every paper around the world extensively reported about the new discovery, with a magazine such as

Science dedicating as many as 23 articles to it in that year alone. Sensationalist reactions to the new discovery included publications linking the new kind of rays to occult and paranormal theories, such as telepathy.

The earliest known experimenter with X-rays was actuary William Morgan. In 1785 he presented a paper to the Royal Society of London describing the effects of passing electrical currents through a partially evacuated glass tube, the first X-ray tube. This work was further extended by Humphry Davy and his assistant Michael Faraday.

In 1876, Eugen Goldstein proved that they came from the cathode, and named them cathode rays (Kathodenstrahlen). Both William Crookes (in the 1880s) and German physicist Johann Hittorf, a co-inventor and early researcher of the Crookes tube, found that paper wrapped photographic plates placed near the tube became unaccountably fogged or flawed by shadows, although they had not been exposed to light. Neither found the cause nor investigated this effect.

In 1877 Ukrainian-born Ivan Pulyui, a lecturer in experimental physics at the University of Vienna, constructed various designs of vacuum discharge tube to investigate their properties. He continued his investigations when appointed professor at the Prague Polytechnic and in 1886 he found that sealed photographic plates became dark when exposed to the emanations from the tubes. Early in 1896, just a few weeks after Röntgen published his first X-ray photograph, Pulyui published high-quality X-ray images in journals in Paris and London. Although Pulyui had studied with Röntgen at the University of Strasbourg in the years 1873–75, his biographer Gaida (1997) asserts that his subsequent research was conducted independently.

X-rays were generated and detected by Fernando Sanford (1854–1948), the foundation Professor of Physics at Stanford University, in 1891. From 1886 to

1888 he had studied in the Hermann Helmholtz laboratory in Berlin, where he became familiar with the cathode rays generated in vacuum tubes when a voltage was applied across separate electrodes, as previously studied by Heinrich Hertz and Philipp Lenard. His letter of January 6, 1893 (describing his discovery as "electric photography") to The Physical Review was duly published and an article entitled Without Lens or Light, Photographs Taken with Plate and Object in Darkness appeared in the San Francisco Examiner.

Starting in 1888, Philipp Lenard, a student of Heinrich Hertz, conducted experiments to see whether cathode rays could pass out of the Crookes tube into the air. He built a Crookes tube (later called a "Lenard tube") with a "window" in the end made of thin aluminum, facing the cathode so the cathode rays would strike it. He found that something came through, that would expose photographic plates and cause fluorescence. He measured the penetrating power of these rays through various materials. It has been suggested that at least some of these "Lenard rays" were actually X-rays.

Hermann von Helmholtz formulated mathematical equations for X-rays. He postulated a dispersion theory before Röntgen made his discovery and announcement. It was formed on the basis of the electromagnetic theory of light. However, he did not work with actual X-rays.

In 1894 Nikola Tesla noticed damaged film in his lab that seemed to be associated with Crookes tube experiments and began investigating this radiant energy of "invisible" kinds. After Röntgen identified the X-ray Tesla began making X-ray images of his own using high voltages and tubes of his own design, as well as Crookes tubes.

In 1895, Thomas Edison investigated materials' ability to fluoresce when exposed to X-rays, and found that calcium tungstate was the most effective substance. Around March 1896, the fluoroscope he developed became the

standard for medical X-ray examinations. Nevertheless, Edison dropped X-ray research around 1903, even before the death of Clarence Madison Dally, one of his glassblowers. Dally had a habit of testing X-ray tubes on his hands, and acquired a cancer in them so tenacious that both arms were amputated in a futile attempt to save his life.

The first use of X-rays under clinical conditions was by John Hall-Edwards in Birmingham, England on 11 January 1896, when he radiographed a needle stuck in the hand of an associate. On 14 February 1896 Hall-Edwards was also the first to use X-rays in a surgical operation. In early 1896, several weeks after Röntgen's discovery, Ivan Romanovich Tarkhanov irradiated frogs and insects with X-rays, concluding that the rays "not only photograph, but also affect the living function".

The first medical X-ray made in the United States was obtained using a discharge tube of Pulyui's design. In January 1896, on reading of Röntgen's discovery, Frank Austin of Dartmouth College tested all of the discharge tubes in the physics laboratory and found that only the Pulyui tube produced X-rays. This was a result of Pulyui's inclusion of an oblique "target" of mica, used for holding samples of fluorescent material, within the tube. On 3 February 1896 Gilman Frost, professor of medicine at the college, and his brother Edwin Frost, professor of physics, exposed the wrist of Eddie McCarthy, whom Gilman had treated some weeks earlier for a fracture, to the X-rays and collected the resulting image of the broken bone on gelatin photographic plates obtained from Howard Langill, a local photographer also interested in Röntgen's work.

In 1901, U.S. President William McKinley was shot twice in an assassination attempt. While one bullet only grazed his sternum, another had lodged somewhere deep inside his abdomen and could not be found. "A worried McKinley aide sent word to inventor Thomas Edison to rush an X-ray

machine to Buffalo to find the stray bullet. It arrived but wasn't used." While the shooting itself had not been lethal, "gangrene had developed along the path of the bullet, and McKinley died of septic shock due to bacterial infection" six days later.

1.2 Benefits of X-ray

The discovery of X-rays and the invention of Computed tomography (CT) represented major advances in medicine. X-ray imaging exams are recognized as a valuable medical tool for a wide variety of examinations and procedures. They are used to, noninvasively and painlessly help to diagnosis disease and monitor therapy; support medical and surgical treatment planning; and guide medical personnel as they insert catheters, stents, or other devices inside the body, treat tumors, or remove blood clots or other blockages. The other general benefit is discussed in chapter four.

1.3 Problem of Research

The crystalline structure of the material is a unique arrangement of atoms in the crystal and these atoms are arranged in a certain way.

Atoms arrangement of within matter is a milestone that may make two substances of the same element quite different. This makes the study of crystalline structure and properties of the material different from other materials. This in itself is difficult to study the crystalline structure of the material.

1.4 Purpose of Research

The aim of this work is to study crystalline structure and materials components by using X-ray diffraction (which gives the density and orientation of crystals) this helps to know the composition of the materials by using Bragg law.

1.5 Review of Previous Results

Many studies carried out on the crystalline structure and knowledge of the components of the material using X - ray diffraction.

Daniel Bellet and Gerard Dolino [1], they used X-ray diffraction to studies of porous silicon, they found that the size and shape of silicon crystallites can be determined from the X-ray diffraction pattern, in particular by using reciprocal space map.

C. J. Cain and A. N. J. Heyn [2], they used X-ray diffraction to studies the crystalline structure of the avian egg shell, from x-ray studies, it is concluded that the avian egg shell is composed of calcium carbonate in the calcite modification. In the main portion (crystalline layer) the calcite occurs in large crystalline areas oriented with the hexagonal axis (17.6 Å) inclined at 28 to 16° from the normal of the shell surface. With respect to the other areas, orientation is present over limited areas. The mammilla layer contains crystallites in entirely random orientation.

Marcello Zocchi and Giuseppe Tieghi [3], they used X-Ray diffraction to studies on catalysis: the crystal structure of di- μ -chloro-bis[(2'-3'- η -exo-3-allylnorborn-2-yl)palladium] and comparison with related complexes relevant to the polymerization of norbornene, they found that the crystal structure of the title complex [a= 11.057(2), b= 7.346(1), c= 6.537(1)Å, α = 80° 32'(2'), β = 104° 31'(2'), γ = 94° 39'(2'), space group P[1 with combining macron]] determined from X-ray diffractometer data and refined by least squares to R= 0.040. Evidence is given for the existence of two factors relevant to catalytic activity: (i) a strong trans influence on the bridging chloride ions favouring dissociation into mononuclear active species; and (ii) a relatively small π component in the metal-olefin bond. These and other structural features are compared with those of similar binuclear complexes.

G. Brauer and others [4], they used X-ray diffraction to study and Characterization of a SiC/SiC composite, it is found that besides its main constituent 3C-SiC the composite still must contain some graphite. In order to better interpret the experimental findings of the composite, a pyrolytic graphite sample was also investigated by slow positron implantation and positron lifetime spectroscopies. In addition, theoretical calculations of positron properties of graphite are presented.

1.6 Content of Research

This search contains several sections. The first section is the general introduction of x-rays. The second section contains how to produce X-rays and how it works. The third section contains the previous studies. The fourth section contains the practical application of x-rays.

Chapter Two

Utilization X-ray and Their Benefits

2.1 Introduction

X-rays make up X-radiation, a form of electromagnetic radiation. Most X-rays have a wavelength ranging from 0.01 to 10 nanometers, corresponding to frequencies in the range 30 petahertz to 30 exahertz (3×10^{16} Hz to 3×10^{19} Hz) and energies in the range 100 eV to 100 keV. X-ray wavelengths are shorter than those of UV rays and typically longer than those of gamma rays. In many languages, X-radiation is referred to with terms meaning Röntgen radiation, after the German scientist Wilhelm Röntgen, who usually is credited as its discoverer, and who had named it X-radiation to signify an unknown type of radiation [5].

2.2 Properties of X-rays

X-ray photons carry enough energy to ionize atoms and disrupt molecular bonds. This makes it a type of ionizing radiation, and therefore harmful to living tissue. A very high radiation dose over a short period of time causes radiation sickness, while lower doses can give an increased risk of radiation-induced cancer. In medical imaging this increased cancer risk is generally greatly outweighed by the benefits of the examination. The ionizing capability of X-rays can be utilized in cancer treatment to kill malignant cells using radiation therapy. It is also used for material characterization using X-ray spectroscopy.

2.3 Production of X-rays

Whenever charged particles (electrons or ions) of sufficient energy hit a material, X-rays are produced.

2.3.1 Production by electrons

X-rays can be generated by an X-ray tube, a vacuum tube that uses a high voltage to accelerate the electrons released by a hot cathode to a high velocity. The high velocity electrons collide with a metal target, the anode, creating the X-rays. In medical X-ray tubes the target is usually tungsten or a more crack-resistant alloy of rhenium (5%) and tungsten (95%), but sometimes molybdenum for more specialized applications, such as when softer X-rays are needed as in mammography. In crystallography, a copper target is most common, with cobalt often being used when fluorescence from iron content in the sample might otherwise present a problem [6].

The maximum energy of the produced X-ray photon is limited by the energy of the incident electron, which is equal to the voltage on the tube times the electron charge, so an 80 kV tube cannot create X-rays with an energy greater than 80 K ev. When the electrons hit the target, X-rays are created by two different atomic processes.

First one is characteristic X-ray emission (X-ray fluorescence), if the electron has enough energy it can knock an orbital electron out of the inner electron shell of a metal atom, and as a result electrons from higher energy levels then fill up the vacancy and X-ray photons are emitted. This process produces an emission spectrum of X-rays at a few discrete frequencies, sometimes referred to as the spectral lines. The spectral lines generated depend on the target (anode) element used and thus are called characteristic lines. Usually these are

transitions from upper shells into K shell (called K lines), into L shell (called L lines) and so on.

Secondly, Bremsstrahlung process, this is radiation given off by the electrons as they are scattered by the strong electric field near the high-Z (proton number) nuclei. These X-rays have a continuous spectrum. The intensity of the X-rays increases linearly with decreasing frequency, from zero at the energy of the incident electrons, the voltage on the X-ray tube.

2.3.2 Production by fast positive ions

X-rays can also be produced by fast protons or other positive ions. The proton-induced X-ray emission or particle-induced X-ray emission is widely used as an analytical procedure. For high energies, the production cross section is proportional to $Z_1^2 Z_2^{-4}$, where Z_1 refers to the atomic number of the ion, Z_2 to that of the target atom [7].

2.4 Interaction with matter

X-rays interact with matter in three main ways, through photo absorption, Compton scattering, and Rayleigh scattering. The strength of these interactions depends on the energy of the X-rays and the elemental composition of the material, but not much on chemical properties, since the X-ray photon energy is much higher than chemical binding energies. Photo absorption or photoelectric absorption is the dominant interaction mechanism in the soft X-ray regime and for the lower hard X-ray energies. At higher energies, Compton scattering dominates.

2.4.1 Photoelectric Absorption

The probability of a photoelectric absorption per unit mass is approximately proportional to Z^3/E^3 , where Z is the atomic number and E is the energy of the incident photon. This rule is not valid close to inner shell electron binding energies where there are abrupt changes in interaction probability, so called absorption edges. However, the general trend of high absorption coefficients and thus short penetration depths for low photon energies and high atomic numbers is very strong. For soft tissue, photo absorption dominates up to about 26 Kev photon energy where Compton scattering takes over. For higher atomic number substances this limit is higher. The high amount of calcium ($Z=20$) in bones together with their high density is what makes them show up so clearly on medical radiographs [8].

A photo absorbed photon transfers all its energy to the electron with which it interacts, thus ionizing the atom to which the electron was bound and producing a photoelectron that is likely to ionize more atoms in its path. An outer electron will fill the vacant electron position and produce either a characteristic photon or an Auger electron. These effects can be used for elemental detection through X-ray spectroscopy or Auger electron spectroscopy.

2.4.2 Compton Scattering

Compton scattering is the predominant interaction between X-rays and soft tissue in medical imaging [9]. Compton scattering is an inelastic scattering of the X-ray photon by an outer shell electron. Part of the energy of the photon is transferred to the scattering electron, thereby ionizing the atom and increasing the wavelength of the X-ray. The scattered photon can go in any direction, but a direction similar to the original direction is more likely, especially for high-energy X-rays. The probability for different scattering angles are described by

the Klein–Nishina formula. The transferred energy can be directly obtained from the scattering angle from the conservation of energy and momentum.

2.4.3 Rayleigh Scattering

Rayleigh scattering is the dominant elastic scattering mechanism in the X-ray regime. Inelastic forward scattering gives rise to the refractive index, which for X-rays is only slightly below 1 [10].

2.5 Uses of X-ray

2.5.1 Medical uses

Since Röntgen's discovery that X-rays can identify bone structures, X-rays have been used for medical imaging. The first medical use was less than a month after his paper on the subject [11]. Up to 2010, 5 billion medical imaging examinations had been conducted worldwide. Radiation exposure from medical imaging in 2006 made up about 50% of total ionizing radiation exposure in the United States [12].

2.5.1.1 Projection Radiography

Projection radiography is the practice of producing two-dimensional images using x-ray radiation. Bones contain much calcium, which due to its relatively high atomic number absorbs x-rays efficiently. This reduces the amount of X-rays reaching the detector in the shadow of the bones, making them clearly visible on the radiograph. The lungs and trapped gas also show up clearly because of lower absorption compared to tissue, while differences between tissue types are harder to see.

Projection radiographs are useful in the detection of pathology of the skeletal system as well as for detecting some disease processes in soft tissue. Some notable examples are the very common chest X-ray, which can be used to

identify lung diseases such as pneumonia, lung cancer, or pulmonary edema, and the abdominal x-ray, which can detect bowel (or intestinal) obstruction, free air (from visceral perforations) and free fluid (in as cites). X-rays may also be used to detect pathology such as gallstones (which are rarely radiopaque) or kidney stones which are often (but not always) visible. Traditional plain X-rays are less useful in the imaging of soft tissues such as the brain or muscle.

Dental radiography is commonly used in the diagnoses of common oral problems, such as cavities.

In medical diagnostic applications, the low energy (soft) X-rays are unwanted, since they are totally absorbed by the body, increasing the radiation dose without contributing to the image. Hence, a thin metal sheet, often of aluminium, called an X-ray filter, is usually placed over the window of the X-ray tube, absorbing the low energy part in the spectrum. This is called hardening the beam since it shifts the center of the spectrum towards higher energy (or harder) x-rays.

To generate an image of the cardiovascular system, including the arteries and veins (angiography) an initial image is taken of the anatomical region of interest. A second image is then taken of the same region after an iodinated contrast agent has been injected into the blood vessels within this area. These two images are then digitally subtracted, leaving an image of only the iodinated contrast outlining the blood vessels. The radiologist or surgeon then compares the image obtained to normal anatomical images to determine whether there is any damage or blockage of the vessel.

2.5.1.2 Computed Tomography

Computed tomography (CT scanning) is a medical imaging modality where tomographic images or slices of specific areas of the body are obtained from a

large series of two-dimensional X-ray images taken in different directions. These cross-sectional images can be combined into a three-dimensional image of the inside of the body and used for diagnostic and therapeutic purposes in various medical disciplines [13].

2.5.1.3 Fluoroscopy

Fluoroscopy is an imaging technique commonly used by physicians or radiation therapists to obtain real-time moving images of the internal structures of a patient through the use of a fluoroscope. In its simplest form, a fluoroscope consists of an X-ray source and a fluorescent screen, between which a patient is placed. However, modern fluoroscopes couple the screen to an X-ray image intensifier and CCD video camera allowing the images to be recorded and played on a monitor. This method may use a contrast material. Examples include cardiac catheterization (to examine for coronary artery blockages) and barium swallows (to examine for esophageal disorders).

2.5.1.4 Radiotherapy

The use of X-rays as a treatment is known as radiation therapy and is largely used for the management (including palliation) of cancer; it requires higher radiation doses than those received for imaging alone. X-rays beams are used for treating skin cancers using lower energy x-ray beams while higher energy beams are used for treating cancers within the body such as brain, lung, prostate, and breast [14, 15].

2.5.2 Other uses

- X-ray crystallography in which the pattern produced by the diffraction of X-rays through the closely spaced lattice of atoms in a crystal is recorded and then analysed to reveal the nature of that lattice. A related

technique, fiber diffraction, was used by Rosalind Franklin to discover the double helical structure of DNA [16].

- X-ray astronomy, which is an observational branch of astronomy, which deals with the study of X-ray emission from celestial objects.
- X-ray microscopic analysis, which uses electromagnetic radiation in the soft X-ray band to produce images of very small objects.
- X-ray fluorescence, a technique in which X-rays are generated within a specimen and detected. The outgoing energy of the X-ray can be used to identify the composition of the sample.
- Industrial radiography uses X-rays for inspection of industrial parts, particularly welds.
- Authentication and quality control, X-ray is used for authentication and quality control of packaged items.
- Industrial CT (computed tomography) is a process which uses X-ray equipment to produce three-dimensional representations of components both externally and internally. This is accomplished through computer processing of projection images of the scanned object in many directions.

- Paintings are often X-rayed to reveal underdrawings and pentimenti, alterations in the course of painting or by later restorers. Many pigments such as lead white show well in radiographs.

- X-ray spectromicroscopy has been used to analyse the reactions of pigments in paintings. For example, in analysing colour degradation in the paintings of van Gogh.

- Airport security luggage scanners use X-rays for inspecting the interior of luggage for security threats before loading on aircraft.
- Border control truck scanners use X-rays for inspecting the interior of trucks.
- X-ray art and fine art photography, artistic use of X-rays, for example the works by Stane Jagodič.
- X-ray hair removal, a method popular in the 1920s but now banned by the FDA.
- Shoe-fitting fluoroscopes were popularized in the 1920s, banned in the US in the 1960s, banned in the UK in the 1970s, and even later in continental Europe.
- Roentgen stereophotogrammetry is used to track movement of bones based on the implantation of markers
- X-ray photoelectron spectroscopy is a chemical analysis technique relying on the photoelectric effect, usually employed in surface science.
- Radiation implosion is the use of high energy X-rays generated from a fission explosion (an A-bomb) to compress nuclear fuel to the point of fusion ignition (an H-bomb).

Chapter Three

Materials and crystalline structures

3.1 State of Matter

In physics, a state of matter is one of the distinct forms in which matter can exist. Four states of matter are observable in everyday life: solid, liquid, gas, and plasma. Many other states are known to exist only in extreme situations, such as Bose–Einstein condensates, neutron-degenerate matter, and quark-gluon plasma, which only occur in situations of extreme cold, extreme density, and extremely high-energy color-charged matter respectively. Some other states are believed to be possible but remain theoretical for now.

Historically, the distinction is made based on qualitative differences in properties. Matter in the solid state maintains a fixed volume and shape, with component particles (atoms, molecules or ions) close together and fixed into place. Matter in the liquid state maintains a fixed volume, but has a variable shape that adapts to fit its container. Its particles are still close together but move freely. Matter in the gaseous state has both variable volume and shape, adapting both to fit its container. Its particles are neither close together nor fixed in place. Matter in the plasma state has variable volume and shape, but as well as neutral atoms, it contains a significant number of ions and electrons, both of which can move around freely.

The term phase is sometimes used as a synonym for state of matter, but a system can contain several immiscible phases of the same state of matter.

3.1.1 Solid Matter

Solid is one of the four fundamental states of matter (the others being liquid, gas, and plasma). It is characterized by structural rigidity and resistance to changes of shape or volume. Unlike a liquid, a solid object does not flow to take on the shape of its container, nor does it expand to fill the entire volume available to it like a gas does. The atoms in a solid are tightly bound to each other, either in a regular geometric lattice (crystalline solids, which include metals and ordinary ice) or irregularly (an amorphous solid such as common window glass).

The branch of physics that deals with solids is called solid-state physics, and is the main branch of condensed matter physics (which also includes liquids). Materials science is primarily concerned with the physical and chemical properties of solids. Solid-state chemistry is especially concerned with the synthesis of novel materials, as well as the science of identification and chemical composition [17].

3.1.2 Liquid Matter

Liquid is one of the four primary states of matter, with the others being solid, gas and plasma. A liquid is a fluid. Unlike a solid, the molecules in a liquid have a much greater freedom to move. The forces that bind the molecules together in a solid are only temporary in a liquid, allowing a liquid to flow while a solid remains rigid.

A liquid, like a gas, displays the properties of a fluid. A liquid can flow, assume the shape of a container, and, if placed in a sealed container, will distribute applied pressure evenly to every surface in the container. If liquid is placed in a bag, it can be squeezed into any shape. Unlike a gas, a liquid is nearly incompressible, it occupies nearly a constant volume over a wide range of pressures; it does not generally expand to fill available space in a container

but forms its own surface, and it may not always mix readily with another liquid. These properties make a liquid suitable for applications such as hydraulics.

Liquid particles are bound firmly but not rigidly. They are able to move around one another freely, resulting in a limited degree of particle mobility. As the temperature increases, the increased vibration of the molecules causes distances between the molecules to increase. When a liquid reaches its boiling point, the cohesive forces that bind the molecules closely together break, and the liquid changes to its gaseous state (unless superheating occurs). If the temperature is decreased, the distances between the molecules become smaller. When the liquid reaches its freezing point the molecules will usually lock into a very specific order, called crystallizing, and the bonds between them become more rigid, changing the liquid into its solid state (unless supercooling occurs) [18].

3.1.3 Gas Matter

Gas is one of the four fundamental states of matter (the others being solid, liquid, and plasma). A pure gas may be made up of individual atoms (e.g. a noble gas like neon), elemental molecules made from one type of atom (e.g. oxygen), or compound molecules made from a variety of atoms (e.g. carbon dioxide). A gas mixture would contain a variety of pure gases much like the air. What distinguishes a gas from liquids and solids is the vast separation of the individual gas particles. This separation usually makes a colorless gas invisible to the human observer. The interaction of gas particles in the presence of electric and gravitational fields are considered negligible as indicated by the constant velocity vectors in the image. One type of commonly known gas is steam.

The gaseous state of matter is found between the liquid and plasma states, the latter of which provides the upper temperature boundary for gases. Bounding the lower end of the temperature scale lie degenerative quantum gases, which are gaining increasing attention. High-density atomic gases super cooled to incredibly low temperatures are classified by their statistical behavior as either a Bose gas or a Fermi gas [19].

3.1.4 Plasma Matter

Plasma is a state of matter resembling an ionized gas, though partially ionized plasmas also exist. This ionization makes the plasma affected by electromagnetic fields, which heavily influence how the plasma behaves. It is one of the four fundamental states of matter, and was first described by chemist Irving Langmuir in the 1920s. Unlike the other three states of solid, liquid, and gas, plasma does not freely exist on the Earth under normal surface conditions, and can only be artificially generated by heating neutral gases or by subjecting that gas to a strong electromagnetic field.

Plasma and ionized gases have unique properties and display behaviors unlike those of the other states, and the transition between them are mostly a matter of nomenclature and subject to interpretation. Based on the surrounding environmental temperature and density, partially ionized or fully ionized forms of plasma may be produced. Neon signs or lightning storms are examples of partially ionized plasma, while the interior of the Sun is an example of fully ionized plasma, along with the solar corona and stars.

The positive charge in ions is achieved by stripping away electrons from atomic nuclei. The number of electrons removed is related to either the increase in temperature or the local density of other ionized matter. This also can be accompanied by the dissociation of molecular bonds, though this process is distinctly different from chemical processes of ion interactions in

liquids or the behavior of ions existing in metals. The response of plasma to electromagnetic fields can be usefully employed in many modern technological devices, such as plasma televisions or plasma etching.

Plasma may be the most abundant form of ordinary matter in the universe, although this hypothesis is currently tentative based on the existence and unknown properties of dark matter. Plasma is mostly associated with stars, extending to the rarefied intracluster medium and possibly the intergalactic regions.

3.2 Crystalline Structure

In crystallography, crystal structure is a description of the ordered arrangement of atoms, ions or molecules in a crystalline material. Ordered structures occur from the intrinsic nature of the constituent particles to form symmetric patterns that repeat along the principal directions of three-dimensional space in matter.

The smallest group of particles in the material that constitutes the repeating pattern is the unit cell of the structure. The unit cell completely defines the symmetry and structure of the entire crystal lattice, which is built up by repetitive translation of the unit cell along its principal axes. The repeating patterns are said to be located at the points of the Bravais lattice.

The lengths of the principal axes, or edges, of the unit cell and the angles between them are the lattice constants, also called lattice parameters. The symmetry properties of the crystal are described by the concept of space groups. All possible symmetric arrangements of particles in three-dimensional space may be described by the 230 space groups.

The crystal structure and symmetry play a critical role in determining many physical properties, such as cleavage, electronic band structure, and optical transparency [20].

3.2.1 Unit cell

The crystal structure of a material (the arrangement of atoms within a given type of crystal) can be described in terms of its unit cell. The unit cell is a box containing one or more atoms arranged in three dimensions. The unit cells stacked in three-dimensional space describes the bulk arrangement of atoms of the crystal. The unit cell is represented in terms of its lattice parameters, which are the lengths of the cell edges (a , b and c) and the angles between them (α , β and γ), 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 reference lattice point. Commonly, atomic positions are represented in terms of fractional coordinates, relative to the unit cell lengths. See fig 3.1.

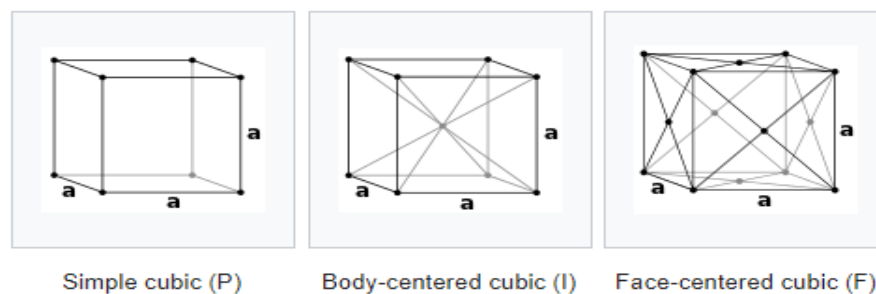


Figure 3.1: shows different unit cells.

The atom positions within the unit cell can be calculated through application of symmetry operations to the asymmetric unit. The asymmetric unit refers to the smallest possible occupation of space within the unit cell. This does not; however, imply that the entirety of the asymmetric unit must lie within the boundaries of the unit cell. Symmetric transformations of atom positions are

calculated from the space group of the crystal structure, and this is usually a black box operation performed by computer programs. However, manual calculation of the atomic positions within the unit cell can be performed from the asymmetric unit, through the application of the symmetry operators described within the International Tables for Crystallography [21].

3.2.2 Miller indices

Vectors and planes in a crystal lattice are described by the three-value Miller index notation. It uses the indices ℓ , m , and n as directional parameters, which are separated by 90° , and are thus orthogonal.

By definition, the syntax (ℓmn) denotes a plane that intercepts the three points a_1/ℓ , a_2/m , and a_3/n , or some multiple thereof. That is, the Miller indices are proportional to the inverses of the intercepts of the plane with the unit cell (in the basis of the lattice vectors). If one or more of the indices is zero, it means that the planes do not intersect that axis (i.e., the intercept is "at infinity"). A plane containing a coordinate axis is translated so that it no longer contains that axis before its Miller indices are determined. The Miller indices for a plane are integers with no common factors. Negative indices are indicated with horizontal bars, as in $\overline{123}$. In an orthogonal coordinate system for a cubic cell, the Miller indices of a plane are the Cartesian components of a vector normal to the plane. See fig 3.2 [22].

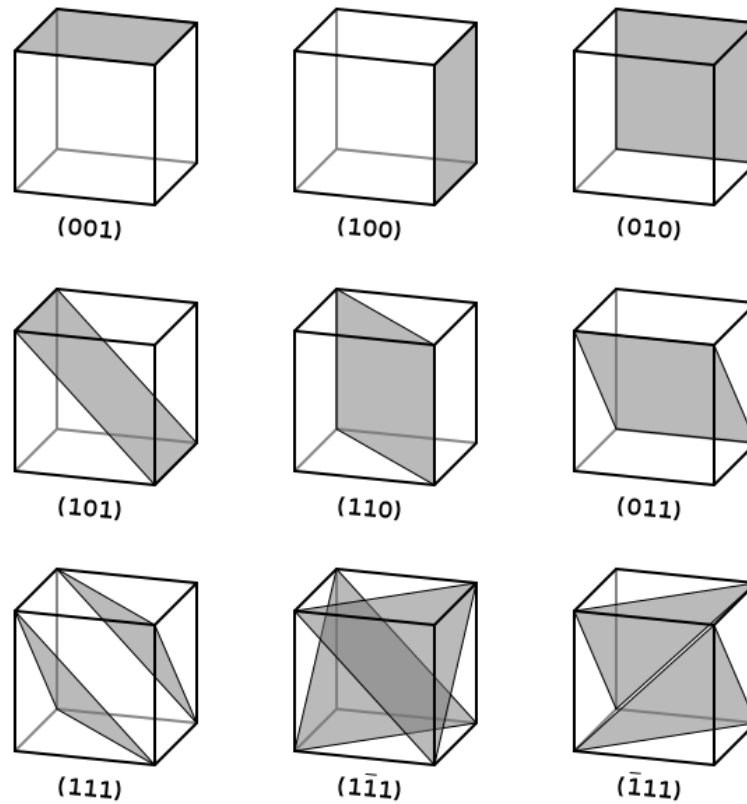


Figure 3.2: Planes with different Miller indices in cubic crystals.

3.2.3 Grain boundaries

Grain boundaries are interfaces where crystals of different orientations meet. A grain boundary is a single-phase interface, with crystals on each side of the boundary being identical except in orientation. The term "crystallite boundary" is sometimes, though rarely, used. Grain boundary areas contain those atoms that have been perturbed from their original lattice sites, dislocations, and impurities that have migrated to the lower energy grain boundary.

Treating a grain boundary geometrically as an interface of a single crystal cut into two parts, one of which is rotated, we see that there are five variables required to define a grain boundary. The first two numbers come from the unit vector that specifies a rotation axis. The third number designates the angle of

rotation of the grain. The final two numbers specify the plane of the grain boundary (or a unit vector that is normal to this plane).

Grain boundaries disrupt the motion of dislocations through a material, so reducing crystallite size is a common way to improve strength, as described by the Hall–Petch relationship. Since grain boundaries are defects in the crystal structure they tend to decrease the electrical and thermal conductivity of the material. The high interfacial energy and relatively weak bonding in most grain boundaries often makes them preferred sites for the onset of corrosion and for the precipitation of new phases from the solid. They are also important to many of the mechanisms of creep.

Grain boundaries are in general only a few nanometers wide. In common materials, crystallites are large enough that grain boundaries account for a small fraction of the material. However, very small grain sizes are achievable. In nanocrystalline solids, grain boundaries become a significant volume fraction of the material, with profound effects on such properties as diffusion and plasticity. In the limit of small crystallites, as the volume fraction of grain boundaries approaches 100%, the material ceases to have any crystalline character, and thus becomes an amorphous solid [23].

3.2.4 Defects and impurities

Real crystals feature defects or irregularities in the ideal arrangements described above and it is these defects that critically determine many of the electrical and mechanical properties of real materials. When one atom substitutes for one of the principal atomic components within the crystal structure, alteration in the electrical and thermal properties of the material may ensue. Impurities may also manifest as spin impurities in certain materials. Research on magnetic impurities demonstrates that substantial alteration of certain properties such as specific heat may be affected by small

concentrations of an impurity, as for example impurities in semiconducting ferromagnetic alloys may lead to different properties as first predicted in the late 1960s. Dislocations in the crystal lattice allow shear at lower stress than that needed for a perfect crystal structure [24].

Chapter Four

Experimental and Methodology

4.1 Introduction

This section discussed the crystalline structure and properties of some materials by using the following devices, X-Ray Diffraction (XRD) and X-Ray Florescent (XRF). The previous studies show that the crystalline structure of the material has a direct effect on their properties such as electrical properties (Band Gap) and mechanical (Material Hardness) and other properties.

In this investigation, the crystalline structure of the materials and the properties of materials investigated in the solid state Labs- College of Science Department of Physics- University of Sudan- Khartoum. Then the components of the materials were studied at the Petroleum Technical Center (PTC) in Khartoum State.

4.2 Apparatus

4.2.1 X-Ray Diffractometer (XRD)

It is important to production X-rays before start a diffraction experiment, so x-rays are electromagnetic radiation similar to light, but with a much shorter wavelength. It is produced when electrically charged particles of sufficient energy are decelerated. In an X-ray tube, the high voltage maintained across the electrodes draws electrons toward a metal target (the anode). X-rays are produced at the point of impact and radiate in all directions- see Fig 4.1. In this work Tubes with Cobalt targets are used to produce their strongest characteristic radiation ($K\alpha_1$) at a wavelength of about 1.79 angstroms ($\lambda=1.7890 \text{ \AA}$).

The goal of using the XRD technique is to identify crystal structure of compounds in materials and characterize the preferred orientation for the layers or crystals (i.e solid samples).

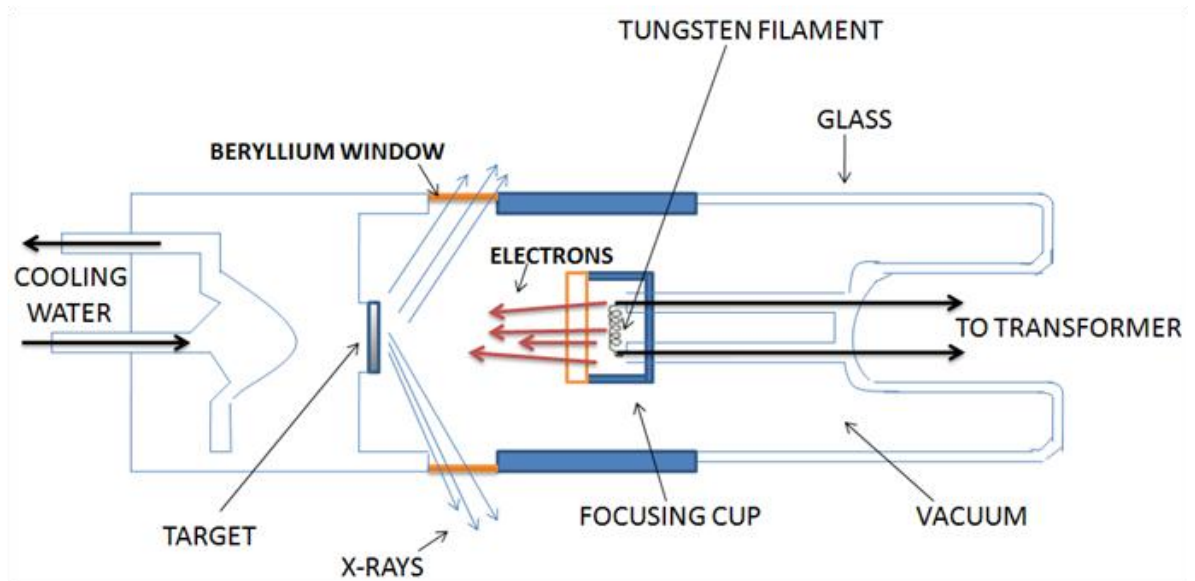


Fig. 4.1: schematic cross section of an x-ray tube.

The basic geometry of an X-ray diffractometer involves a source of monochromatic radiation and an X-ray detector situated on the circumference of a graduated circle centered on the specimen. Divergent slits, located between the X-ray source and the specimen, and Receiving slits, located between the specimen and the detector, limit scattered (non-diffracted) radiation, reduce background noise, and collimate the radiation. The detector and specimen holder are mechanically coupled with a goniometry so that a rotation of the detector through 2θ degrees occurs in conjunction with the rotation of the specimen through θ degrees, see Fig 4.2.

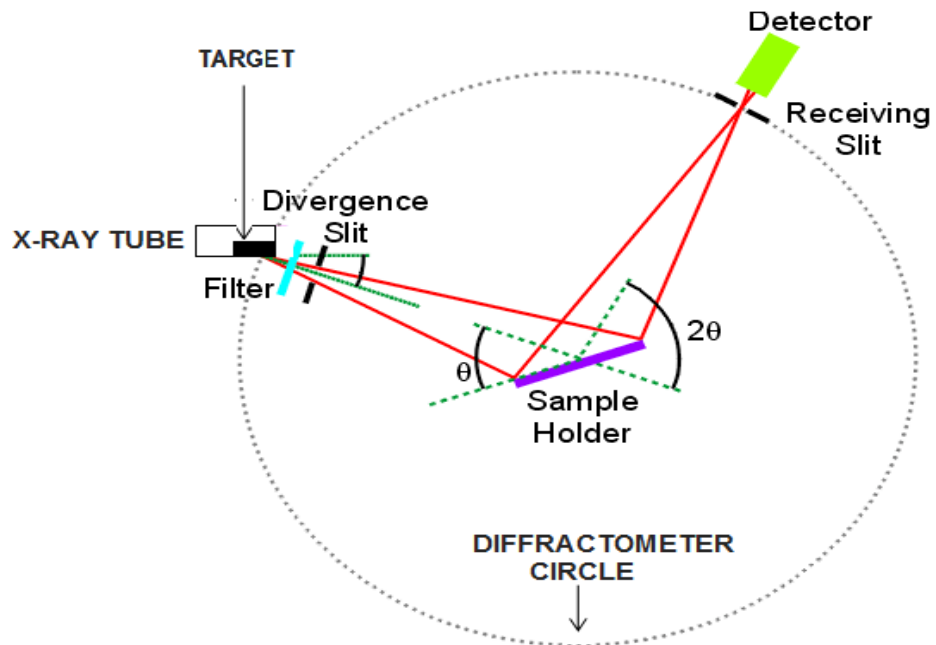


Fig. 4.2: schematic of X-ray diffractometer



Fig 4.3: X-Ray Diffractometer

4.2.2 X-Ray Florescent

XRF is an acronym for x-ray fluorescence, a process whereby electrons are displaced from their atomic orbital positions, releasing a burst of energy that is characteristic of a specific element. This release of energy is then registered by the detector in then XRF instrument, which in turn categorizes the energies by element. Here is a detailed breakdown of the process; an x-ray beam with

enough energy to affect the electrons in the inner shells of the atoms in a sample is created by an x-ray tube inside the handheld analyzer, see fig 4.4. The x-ray beam is then emitted from the front end of the handheld XRF analyzer.

The x-ray beam then interacts with the atoms in the sample by displacing electrons from the inner orbital shells of the atom. This displacement occurs as a result of the difference in energy between the primary x-ray beam emitted from the analyzer and the binding energy that holds electrons in their proper orbits; the displacement happens when the x-ray beam energy is higher than the binding energy of the electrons with which it interacts. Electrons are fixed at specific energies in their positions in an atom, and this determines their orbits. Additionally, the spacing between the orbital shells of an atom is unique to the atoms of each element, so an atom of potassium (K) has different spacing between its electron shells than an atom of gold (Au), or silver (Ag), etc.

The phenomenon is widely used for elemental analysis and chemical analysis, particularly in the investigation of metals, glass, ceramics and building materials. The goal of use this technique is to know all components that in the material (Samples).



Fig 4.4: Handheld X-Ray fluorescence

4.3 Theoretical

Bragg diffraction or Bragg law for X-rays, this diffraction occurs when the wavelength of the electrical radiation or the wavelength of elementary particles is similar to the distance of the atoms in the crystal, where the waves are reflected from layers occupied by atoms and produce a constructive interference according to the law Bragg.

In the crystal, the falling waves are reflected on several layers, separated by the same distance (d) – see fig 4.5. When they interfere, the phase between them remains constant where the path of each wave is equal to the number (n) of the wave length (λ) and the path difference is divided between the two interfering waves produce a constructive interference with the following law

$$2d \sin \theta = n\lambda \quad (4.1)$$

Where:

n : The order of diffraction

d : The interplanar spacing.

θ : Angle of the diffracted beam from a crystal based on the constructive interference of scattered waves, this means the rays are diffracted only when the reflections from the parallel planes of atoms are in phase.

λ : wavelength of X-Ray.

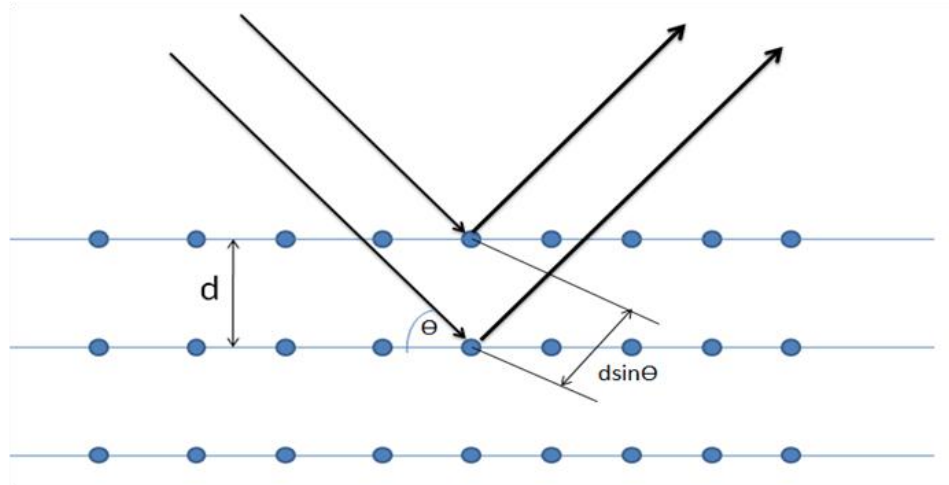


Figure. 4.5: Two beams with identical wavelength and phase approach a crystalline solid and are scattered off two different atoms within it.

4.4 Methodology

In this work, three different samples were used and examined by X-Ray Diffraction (XRD) and X-Ray fluorescent (XRF), the results shows Miller's index for the samples (crystal orientation) and XRF used to study the component of elements of the samples.

XRD used to study the crystalline structure of samples and their properties; one of the samples is placed in the XRD device, which registers the diffraction spectrum by electronic detectors and a computer. Sample is placed in the center of rotary sample holder and exposed to a mono wavelength of X-ray; furthermore, the sample is rotated at a fixed speed around its axis. Some data entered to XRD device such as wavelength (λ), time (t) and current (I), the device drawn the spectrum of dispersion (intensity of radiation - the angle of Bragg), representing each peak dispersion reflection Bragg. This experiment repeated for the three samples in the same way to study its crystalline structure, while XRF device used, which showed the components of each sample.

Handheld XRF Spectrometer used in this study, this device is an instrument used for elemental analysis of a variety of different materials. Samples placed inside the device and X-rays waves bombarded on them, then the device showed the components of the sample in a small screen as shown in figure 4.4 at above paragraph.

4.5 Results

X-ray diffractometer used in this study as mentioned above, results of this device listed below.

Table 4.1: X-ray diffraction for first sample shows Bragg angles and intensities.

β°	$R_0(1/s)$
0	1039.43
1	5791.63
2	10.93
3	22.23
4	14.83
5	14.83
6	19.30
7	28.30
8	15.90
9	13.63
10	11.79
11	10.93
12	10.50
13	14.73
14	10.43
15	12.50
16	8.33
17	8.43
18	8.00
19	6.60
20	8.07
21	6.77
22	6.87
23	7.17
24	7.50

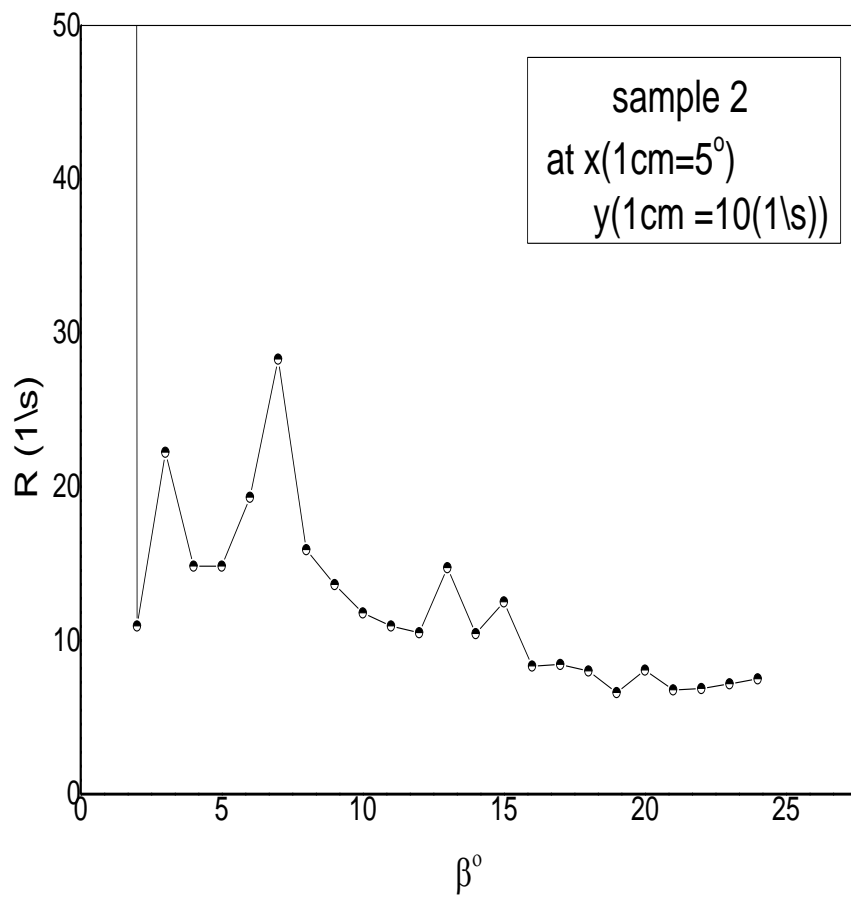


Figure 4.6: X-ray pattern for sample number one.

Table 4.2: X-ray diffraction for second sample shows Bragg angles and intensities.

β°	$R_0(1/s)$
0	0.1
1	0.43
2	0.27
3	0.30
4	0.37
5	0.13
6	0.13
7	0.10
8	0.43
9	0.27
10	0.13
11	0.23
12	0.20
13	0.23
14	0.30
15	0.20
16	0.10
17	0.20
18	0.23
19	0.27
20	0.07
21	0.37
22	0.13
23	0.27
24	0.17

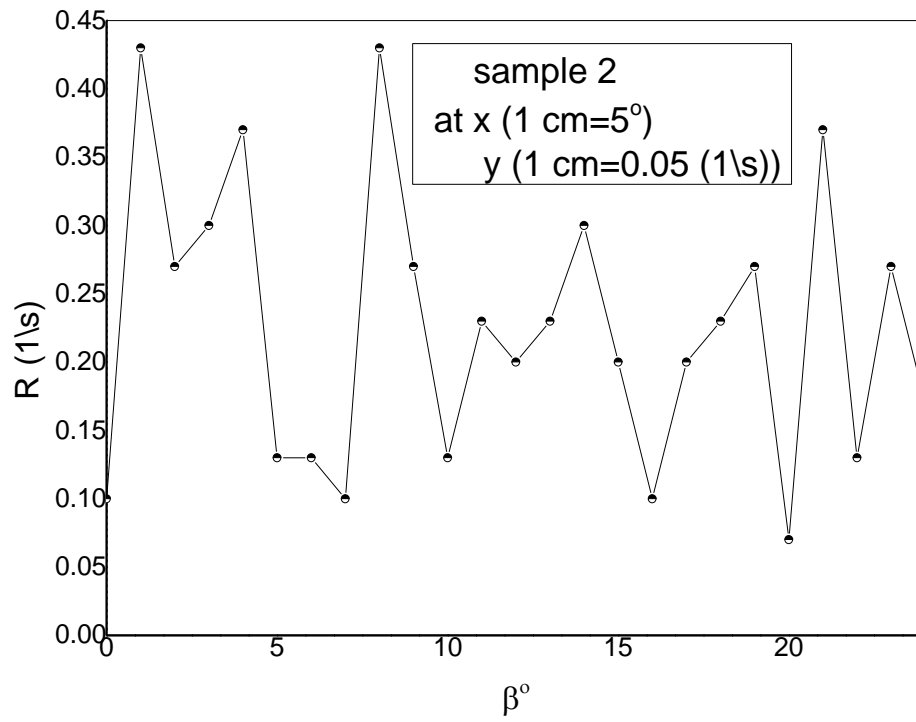


Figure 4.7: X-ray pattern for sample number two.

Table 4.3: X-ray diffraction for third sample shows Bragg angles and intensities.

β°	$R_0(1/s)$
0	540.27
1	151.53
2	0.37
3	0.43
4	0.43
5	0.40
6	0.40
7	0.53
8	0.30
9	0.23
10	0.37
11	0.33
12	0.23
13	0.43
14	0.47
15	0.27
16	0.47
17	0.50
18	0.27
19	0.40
20	0.40
21	0.50
22	0.30
23	0.33
24	0.67

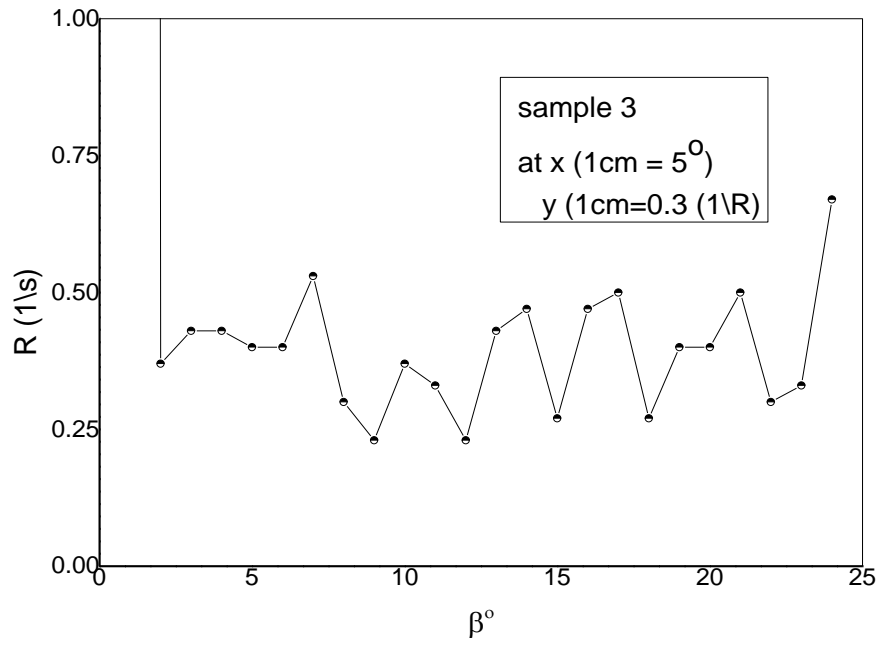


Figure 4.8: X-ray pattern for sample number three.

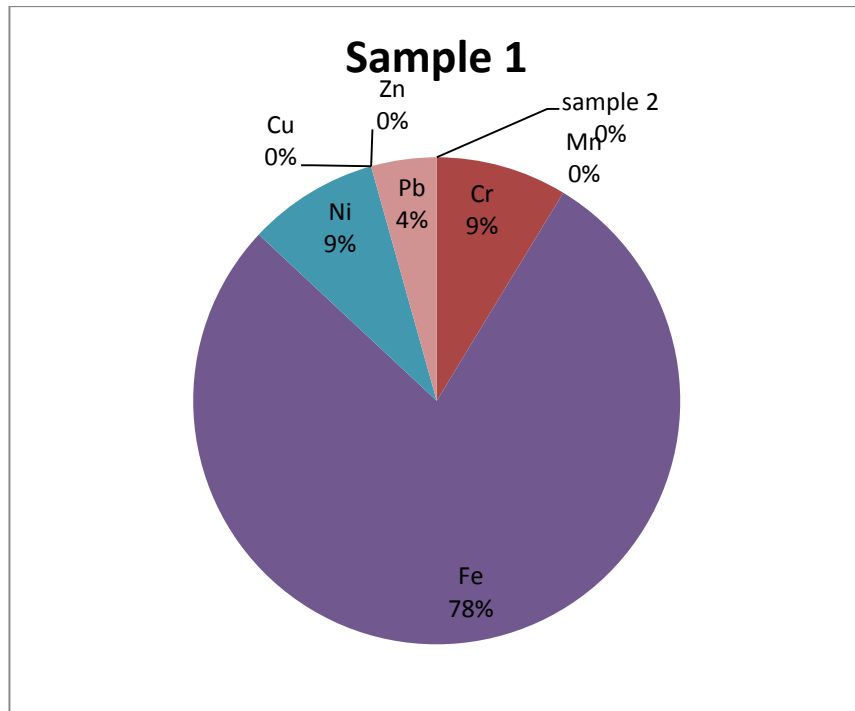


Figure 4.9: XRF shows the percentage of component for sample (Mica).

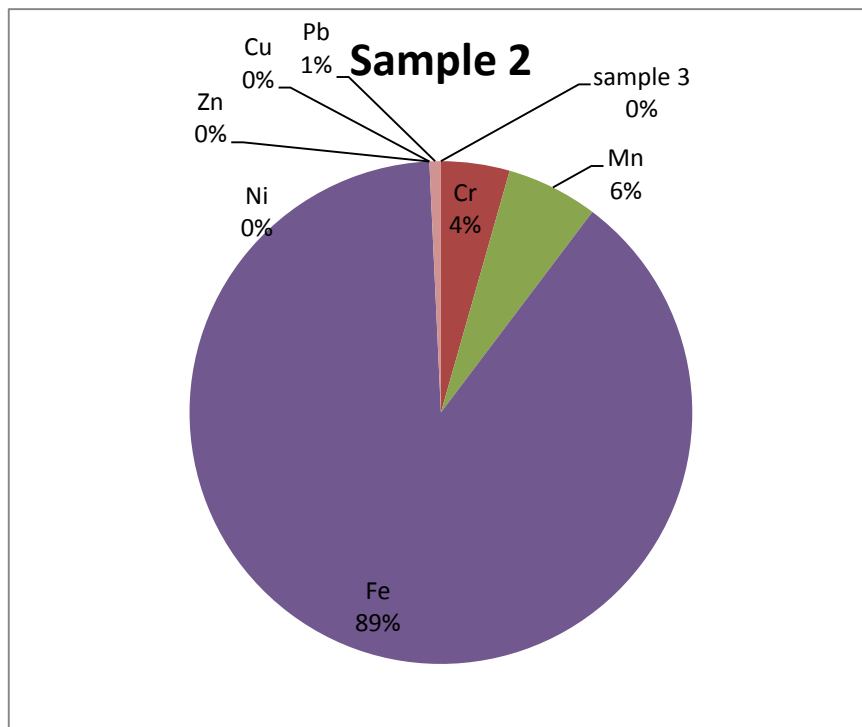


Figure 4.10: XRF shows the percentage of component for sample (Ceramics).

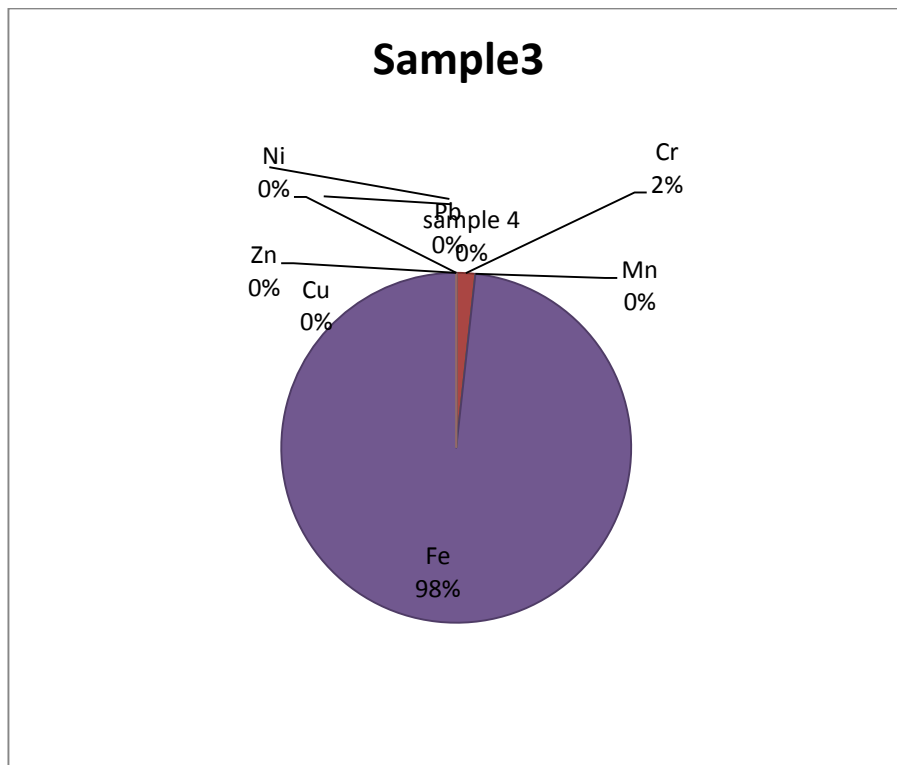


Figure 4.11: XRF shows the percentage of component for sample (Chromium).

4.6 Discussion

In this work, X-Ray diffraction studied for three samples with X-ray diffract meter. The graphs above explained the relationship between the angle of Bragg and the intensity, so the angle selected according to the highest intensity in the above diagram. As the wavelength ($\lambda = 1.8 \text{ nm}$), the interplanar spacing (d) calculated through the Bragg law

$$d = \frac{\lambda}{2 \sin \theta}$$

It found that (d) for the first sample equal $1.3698(\text{nm})$, which is same for the standard data that showed this sample is Mica, while (d) for the second sample is equal to $0.9096(\text{nm})$, then is identical to the ceramics according to

standard data. The interplanar spacing (d) for the third sample showed the Chromium elements.

Through Bragg diffraction, the crystalline structure of the material was known by knowing the interplanar spacing (d) for each sample.

Handheld X-Ray Fluorescence Spectrometer (XRF) used and shown that the first sample is composed of Chromium 9%, Palladium 4% and Nickel 9%, while for the second sample is composed of Manganese 6%, Chromium 4% and Palladium 1%, furthermore the third sample is composed of one component of Chromium 2%.

4.7 Conclusion

The crystalline structure and components of different samples have been investigated using XRD and XRF. The interplanar spacing (d) calculated through the Bragg law, it found that (d) for the first sample equal 1.3698, which is same for the standard data that showed this sample is Mica, while (d) for the second sample is equal to 0.9096 then is identical to the ceramics according to standard data. The interplanar spacing (d) for the third sample showed the Chromium elements.

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4.8 Recommendations

As explained above, through X-ray diffraction we have studied the components of the material, but it will not study the surface of the material because the diffraction here occurs at a depth far from the surface, see below figure. In order to study the surfaces of the materials, we must work to minimize the angle of fall of radiation as much as possible and thus can ensure the diffraction of the radiation from the surface.

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