



# Sudan University of Science and Technology College of Graduate studies



# Preparation and Study of the Structural Properties of Yttrium Barium Copper Oxide by X-ray Diffraction

تحضير ودراسة الخصائص التركيبية لأكسيد اليوتيريوم باريوم نحاس بواسطة تشتت الاشعة السينية

# The Thesis Submitted for Partial Fulfillment for the Requirement of M.Sc. in Physics

 $\mathbf{B}\mathbf{y}$ 

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## بسم الله الرحمن الرحيم

## الآية الكريمة

## يقول الله تعالى:

صدق الله العظيم (سورة العلق)

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First, I want to thank Allah for giving us the full health, patience and motivation during my study to complete this project. I would also like to thank my supervisor **Dr. Mahmoud Hamid Mahmoud Hilo** for his patience and support during our study. Also my thanks and gratitude are extend to my family, friend, colleagues for their encouragement and support which in turn make this study possible Finally, a word of appreciation due to **MR. Mohammed Kamal** for his Assistance in applied part of his study.

## **DEDICATION**

I dedicate this research to

To my mother

To my father

My brothers and sisters

To all my friends

## **List of Abbreviations**

Sample	Abbreviation
$T_{\rm C}$	Critical temperature
XRD	X-ray diffraction
UV-Vis	Ultraviolet-visible Spectroscopy
BEC	Bose- Einstein-condition
BCS	Bardeen-cooper-Schrieffer
YBCO	Yttrium Barium Copper Oxide
NT	Neel Temperature
SEM	Scanning Electron Microscope
HDC	Continuous Magnetic Field
LMCO	Compound of Li, Mo, Cr, and Oxygen

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### **Abstract**

The aim of this study is to prepare three samples of the yttrium barium copper oxide (YBCO) compound, which is classified as a superconducting material with percentages of 100%, 90%, and 80% of the yttrium oxide in the compound respectively, in order to determine the effect of increasing or decreasing the percentage of yttrium oxide on the superconductive property.

The samples were prepared by the usual chemical method and then the three samples were tested using X-ray diffraction (XRD).

The results showed that the samples prepared in their initial ratio are consistent with the compound with superconductivity characteristic with some difference in the values and shape of the primitive unit cell for each sample, the study concluded that the X - ray diffraction test confirms that the compound has superconductive properties, but further application tests such as Levitation and the Joseph Son junction are needed to estimate the Accurate value of the critical temperature of the compound.

### المستخلص

هدفت هذه الدراسة الي تحضيرثلاث عينات من مركب اكسيد اليوتيريوم باريوم نحاس والذي يصنف علي انه من المركبات ذات خاصية التوصيل الفايق بنسب 100%، 90%، 80% لاكسيد اليوتيريوم في المركب وذلك بغرض معرفة تاثير زيادة او نقصان نسبة اكسيد اليوتيريوم علي خاصية التوصيل الفائق للمركب، تم تحضير العينات بالطريقة الكيميائية المعتادة ومن ثم اختبار العينات الثلاثة بطريقة حيود الاشعةالسينية(XRD).

بينت النتايج ان العينات المحضرة تتفق في نسبها الأولية مع البنية المرجعية لمركب اكسيد اليوتيريوم باريوم نحاس ذو الخصائص التوصيلية الفائقة مع بعض الأختلاف في قيم وشكل خلية الوحدة لكل عينة.

خلصت الدراسة الي أن اختبار حيود الأشعة السينية يعطي تاكيد علي أن المركب يتمتع بخاصية التوصيل الفائق، ولكن لابد من اجراء مزيد من الاختبارات التطبيقية مثل الطفو ووصلة جوزيف صن لحساب القيمة الحقيقية لدرجة الحرارة الحرجة للمركب.

# CHAPTER ONE INTRODUCTION

### 1.1 Introduction

The phenomenon of superconductivity, in which the electrical resistance of certain materials completely vanishes at low temperatures, is one of the most interesting and sophisticated phenomena in condensed matter physics. It was first discovered by the Dutch physicist Heike Kamerlingh Onnes, who was the first to liquefied helium (which boils at 4.2 Kelvin at standard pressure)[1]. The most important properties of the superconducting are zero resistance, ideal diamagnetism, expulsion of a magnetic field from a superconductor, and magnetic flux quantization and persistent current in the superconducting rings due to all properties mentioned above, superconductors may be projected for an enormous number of applications as powerful Projected for an electromagnets, power transmission, digital circuits and other applications when zero resistance, persistent currents and very sensitive magnetic field are required .promising future application include high performance transformers, Power storage devices, electric power transmission, electric motors and magnetic levitation devices [2]. It has long been a dream of scientists working in the field of superconductivity to find a material that becomes a superconductor at room temperature. A discovery of this type will revolutionize every aspect of modern day technology such as power transmission and storage, communication, transport and even the type of computers we make. All of these advances will be faster, cheaper and more energy efficient. This has not been achieved to date. However, in 1986 a class of materials was discovered by Bednorz and Müller that led to superconductors that we use today on a bench-top with liquid nitrogen to cool them. Not surprisingly, Bednorz and Müller received the Nobel Prize in 1987 (the fastest-ever

recognition by the Nobel committee). The materials we mostly use on bench-tops is Yttrium – Barium – Copper Oxide, or YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7</sub>, otherwise known as the 1-2-3 superconductor, and are classified as high Temperature(Tc) superconductors [3]. It is well known that, in solids, the atomic structure determines the character of chemical bonding and a number of other related physical properties. Even small changes in structure can considerably change electronic properties of a solid. There are different types of crystal structure of layered copper oxide superconductors have been studied. These various structures can be divided into several families depending on the type of packing of a small number of structure elements, that is, Perovskite - like copper–oxygen CuO<sub>2</sub> layers and buffer rock salt or fluorite block. It should be pointed out that besides the type of crystal structure, that is, the long-range order; the properties of copper superconductors also depend strongly on the short-range atomic order, which determines the local charge distribution in the crystal [4].

## 1.2 Research problem

Resistance is one of the problems that face the manufacturing of electronics devices and components, besides the resistant makes very huge damages in electrical circuit, and although it plays an important roles when we use it as an electrical key for some applications, but the exits of resistance in the wires raises the heat and make many problems, so finding a material with very low resistance at room temperature or near to it is one of the sound fields of research.

YBCO is a ceramic compound that plays (till now) a sophisticated role in searching of ideal superconductors so it is good to keep searching about new properties of it especially the zero resistance point at room temperature.

## 1.3 Objectives:

This research aimed to achieve the following objectives.

## 1.3.1 General objective

Synthesis and structural properties investigations of Yttrium Barium Copper Oxide (YBCO) as a superconductor compound.

## 1.3.2 Specific objectives

- Samples preparation of (YBCO)
- Structural test of the samples using XRD analysis

## 1.4 Methodology

This research follows the practical method or experimental that used to prepare the samples of YBCO and then used types the characterizations method of XRD to characterize the samples, that's because the experimental method is the way of getting accurate and valuable results.

## 1.5 Hypothesis

- -This study hypothesize the following
- -Although of many difficulties that faces the research in Sudan, but it can be nice to prepare a compound such as YBCO and test its superconductivity.
- -YBCO can act very well as superconductor if well prepared.

## 1.6 Layout of the research

This research comes in to fore chapter.

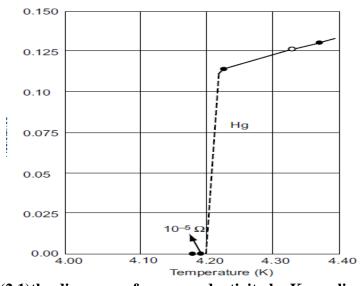
- Chapter one is introduction.
- Chapter two is theoretical back ground and literature review.
- Chapter three is stated for the experimental.
- Chapter four is for the discussion and conclusion.

# CHAPTER TWO THEORETICAL BACKGROUND

## 2.1 Superconductivity

Superconductivity was discovered in 1911 by the Dutch Scientist of Physics in that time Physicist H. Kamerlingh Onnes, only three years after he had succeeded in liquefying helium. During his investigations on the conductivity of metals at low temperature he found that the resistance of a mercury sample dropped to an immeasurably small value just at the boiling temperature of liquid helium. The original measurement is shown in Fig(2.1) Kamerlingh Onnes called this totally unexpected phenomenon 'superconductivity' and this name has been retained since. The temperature at which the transition took place was called the critical temperature Tc. Superconductivity is observed in a large variety of materials but, remarkably, not in some of the best normal conductors like copper, silver and gold, except at very high pressures. This is illustrated in Fig(2.2) where the resistivity of copper, tin and the 'high-temperature' superconductor YBa2Cu3O7 is sketched as a function of temperature [5].

YBa2Cu3O7 is sketched as a function of temperature [5].



Fig(2.1)the discovery of superconductivity by Kamerlingh Onnes

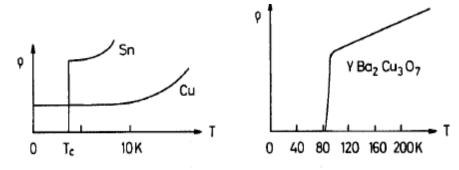


Fig. (2.2)low-temperature resistivity of copper, tin and  $YBa_2Cu_3O_7$ 

1986 the seventy-fifth anniversary the discovery was of superconductivity. Although an enormous amount had been found out about it, superconductivity remained an effect that apparently occurred only at very low temperatures, namely a few degrees above the absolute zero of temperature measured in degrees Kelvin (K). Superconductivity could only be produced under strictly controlled laboratory conditions that precluded widespread applications for it in everyday life. During the 75 years that superconductivity had been known, the critical temperature (Tc) for its onset had only risen slowly from 4.2K, found by Kamerlingh Onnes for the element mercury (Hg), to 23.2K; i.e., 23.2 degrees above the absolute zero of temperature. This highest temperature for Tc was achieved in a thin film of Nb3Ge, an intermetallic compound of the elements germanium (Ge) and niobium (Nb). Superconductivity in this compound was discovered in 1973 and no higher superconducting transition temperature had been found between then and early 1986. Further gloom was cast by some theoretical work suggesting that the highest temperature at which superconductivity could be expected to occur was unlikely ever to exceed 25K. All this changed in April 1986, when Bednorz and Müller submitted an article to the journal Zeitschriftfür Physik in which they described electrical resistance measurements on a complex, ceramic metallic oxide material containing the elements lanthanum (La) – barium (Ba) – copper (Cu) and oxygen (O). They suggested that their data showed

evidence for superconductivity above 30K, a substantial increase over the previous record. Their paper appeared in September 1986. It had an almost immediate impact by December of that year, groups in America and Japan had confirmed and extended the original work. Bednorz and Müller had also carried out further measurements themselves, which greatly strengthened their earlier claims. There then began one of the most amazing and unprecedented sequence of events in physics: many scientists across the world temporarily abandoned their ongoing lines of research in a frantic effort to study these new superconducting ceramics and search for variants with even higher transition temperatures. For a few months, early in 1987, it seemed as if no week went by without the announcement of a further increase in the transition temperature Tc. The subsequent Nobel Prize for the work of Bednorz and Müller paid tribute to those exciting events: the citation commented "This discovery is quite recent – less than two years old – but it has already stimulated research and development throughout the world to an unprecedented extent".

At the beginning of March 1987, a paper appeared in the prestigious journal Physical Review Letters from two groups working together in the United States of America announcing the discovery of a superconductor with a transition temperature for the onset of superconductivity of 93K. This finding had also been made in a ceramic metal oxide, which in this case contained the elements yttrium (Y) – barium (Ba) – copper (Cu) and oxygen (O). The discovery of this new superconductor created enormous excitement because its transition temperature for the onset of superconductivity was above the normal boiling point of liquid nitrogen (77K). Existence of superconductivity in this much higher temperature range greatly enhanced the prospects for widespread applications: an industry based on liquid nitrogen as a refrigerant would be considerably cheaper and easier to operate than one based on the previously known

superconductors, which required working with expensive and difficult helium technology. This second major breakthrough within the space of a few months had important implications for the future direction of several branches of engineering and technology. The increase in the superconducting transition temperature during the 75 years since the initial discovery in 1911 is shown in Fig (2.3)[6].

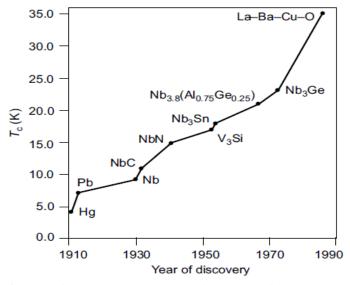


Fig (2.3) plot of the highest superconducting transition temperature obtained against the year for the period between the discovery of superconductivity in 1911 and the advent of high temperature superconductors in 1986.

#### 2.2 Type of Superconductors

A discovery of enormous practical consequences was the finding that there exist two types of superconductors with rather different to magnetic fields. The elements lead, mercury, tin, and others are called 'type I' superconductors. They do not admit a magnetic field in the bulk material and are in the superconducting state provided the applied field is below a critical field Hc that is a function of temperature. All superconducting alloys like lead-indium, niobium-tin, and the element niobium belong to the large class of 'type II' superconductors. They are characterized by two critical fields, Hc1and Hc2 [7].

## **2.3 BCS Theory (Bose-Einstein Condensation)**

An important question concerning the microscopic mechanisms of high-Tc superconductivity is: how the electrons are paired to form the Cooper pairs that are necessary for the condensation of the superconducting state? An answer to this question might be provided by the following hypothesis: the superconducting state arises from a Bose-Einstein condensation (BEC) of existing paired electrons that jump to occupy the double empty 3d levels mentioned in the previous section. Because these electrons were previously paired in atoms or in ions, it is not necessary to assume external interactions (with phonons or other bosons) to account for the pairing energy P of these paired electrons. Another question to analyze is: are these paired electrons in the spin-singlet or in the triplet state? By our hypothesis, these electrons were just paired in atoms or in ions; therefore, these existing pairs are in the spin-singlet state. This singlet state hypothesis is confirmed by Knight Shift experiments (Scalapino, 1995). Is BEC possible in oxide superconductors? According to (Chakraverty et al., 1998) BEC is impossible in oxide superconductors. However, we want to show that BEC in oxide superconductors cannot be ruled out. Initially it is convenient to clarify some concepts regarding BEC. It is well known that a collection of particles (bosons) that follows the counting rule of Bose-Einstein statistics might at the proper temperature and density suddenly populate the measurement of delocalization of a particle must satisfy this condition. We know that  $\lambda dB = h/p$ , where h is Planck's constant and p is the momentum spread or momentum uncertainty of the wave packet. In the other collections ground state in observably large numbers (Silvera, 1997). The average de Broglie wavelength \( \lambda d \) which is a quantum measurement of delocalization of a particle must satisfy this condition. We know that  $\lambda dB =$ h/p, where h is Planck's constant and p is the momentum spread or momentum uncertainty of the wave packet. In the other extreme, for

particles in the zero momentum Eigen state, the delocalization is infinite; i.e., the packet is spread over the entire volume V occupied by the system. It is generally accepted that BEC occurs when the antiparticle separation is of the order of the delocalization λdB (Silvera, 1997). Is BEC possible in oxide superconductors? According to (Chakraverty et al., 1998) BEC is impossible in oxide superconductors. However, we want to show that BEC in oxide superconductors cannot be ruled out. Initially it is convenient to clarify some concepts regarding BEC. It is well known that a collection of particles (bosons) that follows the counting rule of Bose-Einstein statistics might at the proper temperature and density suddenly populate the collections ground state in observably large numbers (Silvera, 1997). The average de Broglie wavelength \( \lambda dB \) which is a quantum extreme, for particles in the zero momentum Eigen state, the delocalization is infinite; i.e., the packet is spread over the entire volume V occupied by the system. It is generally accepted that BEC occurs when the interparticle separation is of the order of the delocalization  $\lambda dB$  (Silvera, 1997).

The thermal de Broglie wavelength  $\lambda dB$  is a measure of the thermodynamic uncertainty in the localization of a particle of mass M with the average thermal momentum. Thus,  $\lambda_{dB}$  is given by (2.1)

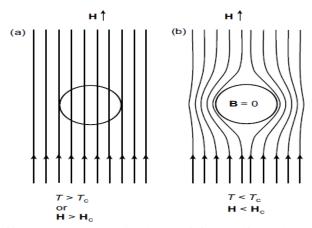
$$\lambda_{\rm dB} = \frac{h}{(3MkT)^{\frac{1}{2}}} (2.1)$$

Where k is Boltzmann's constant. Equation (2.1) shows that at a certain low temperature Tor/and for a small mass M,  $\lambda dB$  may be spread over great distances. In order to determine the critical temperature Tc at which the addition of more particles leads to BEC it is sufficient to calculate a certain critical density n = N/V, where N is the number of bosons. This calculation is performed using Bose-Einstein statistics; according to (Silvera, 1997) and considering  $M = 2m^*$ , where  $m^*$  is the effective mass of the electron, we obtain  $Tc = 3.31h2n2/3/(4\pi2kM)$  (2.2), the first application of BEC

theory to explain 4He super fluidity was realized in 1938 (London, 1938). In an important paper (Blatt, 1962), the BEC approach has been extended to give the same results predicted by BCS theory. Thus, it is reasonable to conclude that the conventional n-type superconductivity in metals (explained by BCS theory) is a special case that can also be considered as a phenomenon of BEC of Cooper pairs [8].

#### 2.4 Meissner Effect

In 1933, Walther Meissner and Robert Ochsenfeld, working in Berlin, made a surprising and fundamental discovery in the magnetic behavior of a superconductor, all materials show some type of magnetic behavior but that found in a superconductors unique and quite remarkable. Meissner and Ochsenfeld observed that when pure tin is cooled in the presence of a magnetic field, on reaching its superconducting transition temperature, the magnetic flux is suddenly completely expelled from its interior, as shown in Fig (2.4). This discovery later became known as the Meissner effect. Since diamagnetism is a measure of the ability of a material to shield its interior from an applied magnetic field, the complete exclusion of flux means that a superconductor is a perfect diamagnetic [9].



Fig(2.4) Meissner effect The magnetic lines of force (flux lines) in and around a metal, which goes superconducting: (a) In the normal state above the critical temperature Tc or when the applied magnetic field is greater than the critical field  $H_c$  (b) When the sample is cooled below Tc, the metal becomes superconducting and the magnetic field is completely expelled from the superconductor; hence the flux density B inside is zero.

## 2.5 Levitation in superconductivity

Faraday's laws of electromagnetism tell us that when the magnet is moved towards the superconducting sample, a persistent or screening current is induced in the surface layer of the superconductor. This current in turn generates a magnetic field, which is exactly equal and opposite in sense to that of the magnet itself. Their exactly opposing magnetic field causes the magnet and the superconductor to repel each other mutually with a force sufficient to result in levitation of the magnet, which stays floating above the superconductor. Since the superconducting current remains constant in the zero resistance material, the magnet will continue to float as long as the sample is kept below its superconducting transition temperature. Inside the bulk of the superconductor itself the magnetic flux is zero – as required by the Meissner effect [9].



Fig (2.5) Levitation, induced by the persistent surface current and the Meissner effect, of a powerful, small samarium—cobalt permanent magnet above a YBCO polycrystalline sample produced by students in the Department of Physics of the University of Bath.

## 2.6 Structure of copper-oxide

The structure of quaternary copper-oxide compounds with the general formula (Ln1-xMx)n+1CunO3n+1-m(where Ln is a trivalent rareearth ion RE or Y, and M is a divalent alkaline ion, Ba, Sr, or Ca) can be characterized by the packing of CuO6 octahedrons and an ordered system of oxygen vacancies. The number of copper-oxygen planes is defined by

the quantity n = 1, 2, ... For n = 1, we have the layered perovskite structure of K2NiF4, while for  $n\rightarrow\infty$ we get a cubic perovskite ABO3 (see Fig((2.1). The number of oxygen vacancies is characterized by the quantity m describing the multiplicity of copper coordination. Since copper readily allows the four-fold coordination CuO4 in a plane and the five-fold coordination CuO5in a pyramid besides the six-fold coordination CuO6 in an octahedron, a large number of perovskite like structures with oxygen deficiency appear. In these compounds, the coppers usually in the state Cuv+ with a formal valence  $2 \le v \le 2.4$ , so that the number m turns out to be related to the concentration x of divalent ions M. Among various copper-oxide structures, only few of them that have regular CuO2 planes with a particular degree of oxidation become superconducting. The most studied compounds are La1-xMxCuO4 and YBa2Cu3O7-y[10].

### $2.7YBa_2Cu_3O_{7-\nu}$ (YBCO)

The frenetic search for superconducting cuprite's with high transition temperatures quickly led to the discovery of YBCO with a transition temperature of around 93K by Chu and colleagues at Houston. Considerable attention has been focused on YBCO because it is the first superconductor to be discovered which can be used in liquid nitrogen (boiling point 77K). The economic advantage of this can be seen by noting that the difference in price between liquid nitrogen and liquid helium. On top of this, a helium system is inherently complex and prone to breaking down: a technology based on liquid nitrogen as a refrigerant is enormously attractive and is now in place. YBCO remains the best known and most widely used of the high temperature superconductors. One reason for its popularity is that it is possible to make a polycrystalline sample of this material by a reasonably straightforward and non-hazardous process [11]. The superconducting phase in this compound has the layered perovskite structure YBa2Cu3O7-y(YBCO) with a deficit in oxygen.

Thus, within one year, the temperature of the superconducting transition increased several times as compared to the value Tc = 23K, the record known in 1973. It is very important that Tc in the new copper-oxide superconductors exceeds the boiling point of liquid nitrogen which is a criterion defining a true high-temperature superconductor [12].

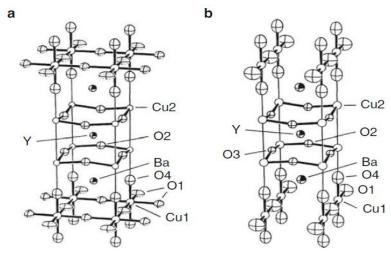
## 2.8 Structure properties of YBCO

Most high temperature superconductors have a tetragonal or orthorhombic structure. The original compound YBa2Cu3O7-ycan be synthesized in two structural modifications depending on temperature and oxygen content y. The first is the orthorhombic phase Pmmm(D1 2h). The second is the tetragonal phase P4/mmm(D7 4h). The elementary cells of these structures with one formula unit are shown in Figs 2.6a,b. The main structural parameters in the orthorhombic phase (Fig2.6b) (in  $^{\circ}$ A) at room temperature and y \_ 0 are the following. The lattice constants are a = 3.828, b = 3.888, c = 11.65.

The lengths of the bonds are Cu1-O1\_Cu1-O4 = 1.94, Cu2-O2 = 1.92,Cu2-O3 = 1.96, Cu2-O4 = 2.3. The length of the four Cu-O bonds for the four oxygen ions nearest to the copper both in the plane Cu2-O2, O3 and in the chains Cu1-O1, O4 are approximately the same and correspond to the lengths of bonds in the CuO2 plane for LMCO compounds. The distance between the copper ion in the plane and the apex oxygen, Cu2-O4, (as the lattice constant c) varies strongly, as the oxygen content decreases under the transition into the tetragonal phase (see Fig 2.7). In the tetragonal phase (Fig 2.6a), oxygen positions O2 and O3 become equivalent with equal bond length Cu2-O2 and Cu2-O3, while remaining structural parameters are close to those in the orthorhombic phase. The orthorhombic phase is observed at low temperatures for an oxygen content  $= 7-y \ge 6.4$ . The transition to the tetragonal phase occurs

at temperatures  $T \ge 500$ °C when oxygen content starts to decrease together with disordering of oxygen ions in the Cu1–O1 plane (Fig 2.6a). It is seen that YBCO has a typical layered perovskite-like structure with two CuO2 planes separated by an oxygen free layer of Y ions which are coupled by the buffer layers Ba–O4–Cu1–O1–Ba–O4. The oxygen O2 and O3 are strongly coupled with Cu2 in the CuO2 planes, unlike the weakly coupled oxygen O1 in theCu1–O1 chains. Upon heating above 500°C, the latter oxygen ions diffuse away from the sample. This enables the oxygen content to be smoothly varied from x = 7 (y = 0) to x = 6 (y = 1), when all oxygen ions O1 in chains have been removed out of the compound. In the latter case, the tetragonal phase persists up to low temperature. At intermediate values of x, the structure of the compound depends on the way in which oxygen is removed [13].

The physical properties of YBa2Cu3O7-y-based compounds depend considerably on the oxygen content. The highest value of superconducting temperature, Tc = 92K, is attained at the optimal doping in metallic phase at x = 7 - y = 6.92. With decreasing oxygen content, Togoes down and the metallic phase transforms into the semiconducting phase at y \_ 0.6. In the latter phase, long-range AF order appears with a maximum Neel temperature TN 500K at y = 1. The way in which Tc depends on x = 7 - yis determined by the type of sample preparation. In Fig(2.13a), the curve Tc(x) is plotted. The dots correspond to high-temperature quenching and the crosses to the lower-temperature Zr-gettering (solid line). In the latter case, two plateau are observed at Tc = 90K (for 6.85 < y < 7.0) and Tc = 60K(for 6.45 < y <6.65), which are related to the aforementioned two orthorhombic phases OI and OII. Thus, the short-range order in the Cu1-Olchain has an essential effect on the electronic properties of the superconductor. This point to a local nature of the doping of the conducting CuO2planedue to charge (hole) transfer from the Cu1–O1 chains.



Fig(2.6) the structure of  $YBa_2Cu_3O_7-y$  in (a) the tetragonal and (b) the orthorhombic phases

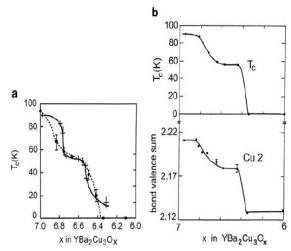


Fig. 2.13 (a) The dependence of Tc on oxygen content x in  $YBa_2Cu_3Ox$  for samples with oxygen removed by high-temperature quenching (dots) and by lower temperature Zrgettering  $(solid\ line)$  and (b) the relation between Tc and the effective copper valence

The dependence of Tc and the lattice parameters of the YBCO compound on the oxygen content have been studied in detail by Cava et al. by applying the lower-temperature Zr-gettering annealing technique. This technique has permitted a number of low-temperature equilibrium phases to be obtained by varying the oxygen content and has revealed a correlation between changes in electronic properties, in particular, Tc, and structural parameters. Fig(2.8a) plots the dependence of the lattice parameters a, b, and c in the orthorhombic phase on the oxygen content x = 7-y [215]. At x = 6.4, under the transition from the orthorhombic (O) to the tetragonal (T)

phase, a considerable increase in the c-axis lattice constant is observed. The length of the out-of-plain bond Cu2–O4 (O4 is the apex oxygen in the CuO5 pyramid, see Fig(2.6), is denoted as O1 in Fig. (2.8b) undergoes equally strong variation, while only a slight variation of the in-plane bond lengths, Cu2–O2, O3, occurs as shown in Fig(2.8b). To find the correlation between the change in structural parameters and electronic properties, a

bond valence sum  $V = ie^{\frac{(x_0 - A_i)}{B_i}}$  has been calculated for various ions.

The sum determines an effective valence of a given ion specified by the parameters Ri and Bi. The effective valence of copper ions in Cu1 chains has turned out to be linear in x. It varies from V = 2.5 at x = 7 to  $V_1 = 1.3$  at x = 6. The effective valence of Cu2 undergoes a downwards jump at the transition from the orthorhombic to tetragonal phase, which is accompanied by a correspondingly sharp variation in the length of the Cu2–O4 bond. Fig(2.13b) demonstrates a correlation between Tc and the effective copper valence. The similar behavior of these functions indicates that Tc is determined by the value of the effective charge in the CuO2 plane. The decrease of Tc from 92 to 60K is due to the transfer of (negative) charge of about 0.03 e from the chains to the plane. The disappearance of superconductivity at x = 6.45 is connected with a further transfer of charge of about 0.05 e to the plane. As the oxygen content further decreases to x =6, the effective charge of the copper in the plane remains constant. The charge transfer from the chains to the plane under oxygen doping was confirmed later in the photoemission experiments.

Thus, detailed structural studies of YBa2Cu3Ox compounds have unambiguously demonstrated the local nature of the charge transfer from CuO chains to CuO2 planes and revealed drastic changes in the electronic properties of the system, including superconductivity, related to the transfer. Cava et al. have noted that for samples with the same oxygen

content, the transition temperature Tc can vary considerably depending on the oxygen ordering in the Cu1–O1 chains. The positive charge transfer (holes) from chain to plane can occur only when two oxygen positions O1, nearest to a copper site Cu1 in the chain O1–Cu1–O1, are filled by oxygen ions. Then, the formal valence of the Cu1 becomes v >2, which produces a hole transfer from the plane to oxygen in chains. It explains why the short-range order of oxygen ions in chains is so important for proper hole doping of CuO2planes [14].

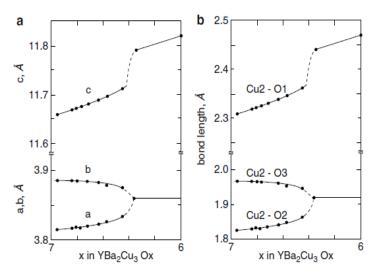


Fig. 2.8 The dependence of (a) lattice constants and (b) copper-oxygen bond lengths  $(Cu_2-O_2, O_3)$ ,  $(Cu_2-O_4)$  in  $YBa_2Cu_3O_x$  on oxygen content x

#### 2.9 X-Ray diffraction.

#### 2.9.1 Background

Solid matter consists out of two types of material: amorphous and crystalline. In an amorphous sample the atoms are arranged in a random way, glasses are an example of amorphous materials. In a crystalline sample the atoms are arranged in a regular or ordered pattern and there is a smallest volume element that, by repetition in three dimensions, can describe the crystal. This smallest volume element is called a unit cell. The dimensions of this unit cell can be described by three axes namely: a, b and c and the angles between the axis are  $\alpha$ ,  $\beta$ , and  $\gamma$ < and =. A schematic diagram of the unit cell is given in Fig (2.9) [15].

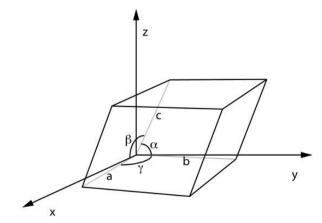


Fig (2.9) A unit cell from a three dimensional lattice

X-ray diffraction (XRD) is a powerful method for the study ofnanomaterial (materials with structural features of at least one dimension in the range of 1-100 nm). The wavelength of X-rays is on the atomic scale, so X-ray diffraction (XRD) is a primary tool for probing structure of nano-materials. XRD offers unparalleled accuracy in the measurement of atomic spacing and is the technique of choice for determining strain states in thin films. The intensities measured with XRD can provide quantitative, accurate information on the atomic arrangements at interfaces. With lab-based equipment, surface sensitivities down to a thickness of ~50 A° are achievable, but synchrotron radiation allows the characterization of much thinner films and for many materials, monoatomic layers can be analyzed. XRD is non-contact and non-destructive, which makes it ideal for in situ studies. Nonmaterial has a characteristic microstructure length comparable with the critical length scales of physical phenomena, giving them unique mechanical, optical and electronic properties. X-ray diffract grams of nonmaterial provide a wealth of information - from phase composition to crystallite size, from lattice strain to crystallographic orientation. The main use of powder diffraction is to identify components in a sample by a search/match procedure. Furthermore, the areas under the peak are related to the amount of each phase present in the sample. In 1919 A.W. Hullgavea paper titled, "A New Method of Chemical Analysis". Here he pointed out that "Every crystalline substance gives a pattern; the same substance always gives the same pattern; and in a mixture of substances each produces its pattern independently of the others. "The X-ray diffraction pattern of a pure substance is, therefore, like a fingerprint of the substance. The powder diffraction method is thus ideally suited for characterization and identification of polycrystalline phases.

## 2.9.2 Theoretical Principles

Fig(2.10) shows the basic features of an X-ray diffract meter, in which the diffraction angle  $2\Theta$  is the angle between the incident and diffracted X-rays. A typical diffraction spectrum consists of a plot of reflected intensities versus the detected angle  $2\Theta$ . The  $2\Theta$ values of the peak depend on the wavelength of the anode material of the X-ray tube. By choosing the right anode and energy of accelerated electrons, a known wavelength and therefore a known energy of X-rays will be generated. Copper X-ray tubes are most commonly used for X-ray diffraction of inorganic materials. For practical applications of X-ray diffraction, we typically want to use x-rays of a single wavelength, i.e. monochromatic radiation to improve experimental results. In general,  $K\alpha$  radiation is used for analytical work while all other radiation ( $K\beta$ , etc.) are removed by means of a nickel filter.

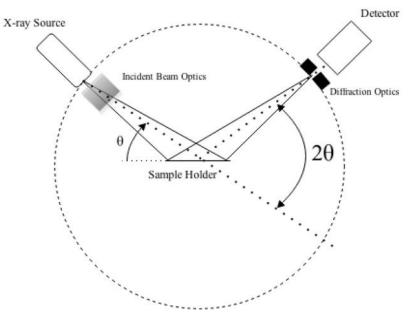


Fig (2.10)Basic features of an X-ray diffractometer

The lattice planes in the simple crystal in Fig (2.11) are separated by a distance d. The Bragg's law relates the wavelength ( $\lambda$ ) of the reflected X-ray, the spacing between the atomic planes (d) and the angle of diffraction ( $\theta$ ) as follows.

 $2d \sin \theta = n \lambda(2.3)$ 

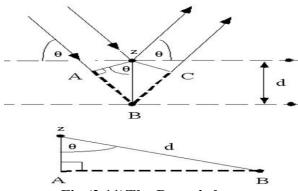


Fig (2.11)The Bragg's law

The angle between the transmitted and diffracted beams willalways be equal to 2Θ. This angle can be obtained readily in experimental situations and the results of X-ray diffraction aretherefore given in terms of 2Θ. It is however very important to remember that the angle that is used in the Bragg's equation must correspond to the angle between the incident radiation and the diffracting plane.

For the first order diffraction, n=1, and knowing  $\theta$  and  $\lambda$ , one can calculate the interplanar spacing d-value for a particular plane. Fig (2.11) shows the information we can get from an idealized diffraction pattern. The first step of X-ray diffraction pattern involves the indexing of XRD peaks. The indexing means assigning the correct Miller indices to each peak of the diffraction pattern. There are three main methods for indexing a diffraction pattern; one of them is comparing the measured XRD pattern with the standard data base [16].

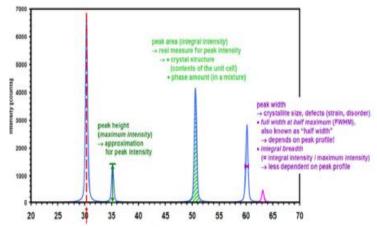


Fig (2.11)Information content of an idealized diffraction pattern

#### 2.9 The Literature Review

Guanhua Chen, Jean-Marc Langlois, YuejinGuo, and William A Goddard study the Superconducting properties of copper oxide high-temperature superconductors (YBaCuO/LaSrCuO/magnon pairing/generalized valence bond calculations/Heisenberg coupling term). Concluded that, equations for the magnon pairing theory of high temperature copper-oxidebased superconductors are solved and used to calculate several properties, leading to results for specific heat and critical magnetic fields consistent with experimental results. In addition, the theory suggests an explanation of why there are two sets of transition temperatures (T, - 90 K and 55 K) for the YBa2Cu3O6, class of superconductors. It also provides an explanation of why La2zSr.CuO4 is a superconductor for only a small range of x (and suggests an experiment to independently test the theory). These results provide support for the magn on pairing theory of high-temperature superconductors. On the basis of the theory, some suggestions are made for improving this material [17]. Tsuyoshi ARIYOSHI and Takanori SAWAI study the Application of Superconductors. In recent years, global warming has become a serious environmental issue. Development efforts are currently underway toward achieving practical application of hightemperature superconducting wires. A high-temperature superconductor has zero electrical resistance at the temperature of liquid nitrogen, so it can reduce the power losses in electrical equipment. The authors have developed a prototype electric vehicle equipped with a motor system that uses bismuth superconducting wire to verify the potential and problems of superconductors. It was verified that the prototype superconducting motor has a torque of 70 Nm, an output of 18 kW and a maximum speed of 70 km/h. The maximum torque can be achieved at low rotations, and therefore a smooth start and acceleration is possible. After six months of test driving, there has been no problem [18].

**Dale R. Harshman, Anthony T. Fiory and John D. Dow,** theory of High-TC Superconductivity: Transition Temperature.

### **Concluded That**

It is demonstrated that the transition temperature (TC) of TC high-superconductors is determined by their layered crystal structure, bond lengths, valency properties of the ions, and Coulomb coupling between electronic bands in adjacent, spatially separated layers. Analysis of 31 high-TC materials (cuprates, ruthenates, rutheno-cuprates, iron pnictides, organics) yields the universal relationship for optimal compounds,KBTCO =  $B/\ell$ , where  $\ell$  is related to the mean spacing between interacting charges in the layers,  $\delta$  is the distance between interacting electronic layers,B is a universal constant and TCO is the optimal transition temperature (determined to within an uncertainty of +/- 1.4 K by this relationship). Non-optimum compounds, in which sample degradation is evident, e.g. by broadened superconducting transitions and diminished Meissner fractions, typically exhibit reduced TC< TCO. It is shown that may be obtained from an average of Coulomb interaction forces between the two layers[19].

Serkan ALAG"OZstudy the Production of YBCO Superconductor Sample byPowder-In-Tube Method (PITM); and Effect of Cd and Ga Doping on the System.

Concluded That In his study, YBa2Cu3O7- $\delta$ and Y0.5X0.5 Ba2 Cu3O7- $\delta$ (x = Ga, Cd) compounds are produced by Powder-In-Tube Method and electrical and physical characteristics of those samples were

investigated. Following 3, 5 and 9 tons pressure applied to three sample groups (YBa2Cu3O7-δ, Y0.5 Cd0.5 Ba2 Cu3O7-δ,Y0.5 Ga0.5 Ba2 Cu3O7-δ), samples were examined via X-Ray diffraction, resistivity and Scan Electro Microscope measurement. Considerable changes in Crystallographic properties of the YBCO samples, produced by using Powder- In-Tube Method for different pressure values, were observed. Under different magnetic fields conditions, resistance of the YBCO samples was measured and it was seen that samples had very pure and homogenous structure. Ga doping into YBCO composition had negative effect on superconducting phases. Cd doping of YBCO induced a more positive effect on the system than Ga doping. Contrary, Cd doping negatively affected electrical properties of the YBCO system.

PawelPeczkowski, PiotrSzterner, Zbigniew. Jaegermann1,MarcinKowalik, Ryszard Zalecki, and Wieslaw Marek Woch,( Journal of Superconductivity and Novel Magnetism), Effects of Forming Pressure on Physicochemical Properties of YBCOCeramics.

#### **Concluded That**

The solid-state synthesis of high-temperature superconducting YBa2Cu3O7-x. Tests were carried out onsamples formed at different pressures (200, 400, 600 and 800 MPa) before being annealed under pure oxygen. The X-ray diffraction method showed that, regardless of the forming pressure, the samples contain about 97 wt.% of Y-123 phase. SEMimages showed a polycrystalline structure of samples of similar grain size and number of pores (intergranular spaces). The values of critical temperatures (Tc°), determined from magneto resistance measurements, are about 91.5 K for all samples, and the Tc° temperatures do not depend on sample-forming pressure. Magneto resistance measurements have shown that samples formed with higher pressures exhibit smaller changes in Tc° and superconducting transition width \_T due to the influence of the HDC

magnetic field, than the samples formed with lower pressures. Values of specific resistance determined by the use of van der Pauw method at 300 K is about 2 cm for all samples. The critical temperatures (T intra c ) of grains and critical current densities at 77 K were determined from AC magnetic susceptibility measurements, and they are about 91.6 K and 400 A cm-2, respectively [21].

# CHAPET THREE EXPERIMENTAL

## 3.1 Materials

- Yttrium oxide  $(Y_2O_3)$  Wight powder
- Barium Carbonate (BaCO<sub>3</sub>) -
- Copper oxide (CuO) black powder
- Diluted sulfuric acid
- Distilled water

## **3.2 Devices (apparatus)**

Marble Mortar

500 ml crucible





1500 C<sup>0</sup> Carbolated oven



### Sensitive balance





## 3.3-Method

To obtain the desired superconducting compound, each of Y2O3(0.151gm), BaCO3 (0.529 gm), CuO(0.320 gm) were considered as initial weights equivalent to 1 gm for the whole sample. There were three samples prepared in order investigate the present of Yttrium in the compound. To increase the quantity of the sample, each of the starting materials were multiplied by 3, and as the results the new calculated weights will be according to the following

The blow weights in gram were obtained using the equation (3.1) [22].

$$\frac{1}{2}Y_2O_3 + 2BaCO_3 + 3CuO \rightarrow YBa_2Cu_3O_7 + 2CO_2 \uparrow (3.1)$$

Sample (1)

$$Y_2O_{3n}(0.151gm)*3 = 0.453gm$$
 (3.1)  
 $BaCO_3(0.529 gm)*3 = 1.587gm$   
 $CuO(0.320 gm)*3 = 0.96gm$ 

## Sample (2)

Y2O3 
$$(0.1359 \text{ gm})^*3 = 0.4077 \text{gm}$$
 (3.2)  
BaCO3  $(0.529 \text{ gm})^*3 = 1.587 \text{gm}$   
CuO  $(0.320 \text{ gm})^*3 = 0.96 \text{gm}$   
Sample (3)  
Y2O  $(0.1208 \text{ gm})^*3 = 0.3624 \text{gm}$  (3.3)  
BaCO3  $(0.529 \text{ gm})^*3 = 1.587 \text{gm}$   
CuO  $(0.320 \text{ gm})^*3 = 0.96 \text{gm}$ 

In the way of preparing the sample, the above weights were achieved using a sensitive balance with atmosphere insulating glass. In the beginning, the Mortar was washed using the distilled water, and diluted sulfuric acid by 40%. The washing process has been done in sequence, as the tool were washed first using tap water, followed by immersing into the diluted sulfuric acid, then kept to dry and washed for second time by using the distilled water. Tools were eventually washed by acetone to guaranty the removal of any unwanted contaminations.

Beginning with the first sample (100% present of Y<sub>2</sub>O<sub>3</sub>), the dry mix is ground with clockwise gentle movement for 30 mints, until a gray mixed powder is obtained. The powder is carefully transferred to high temperature resistance crucible, and placed into Carbolated oven for 1 hour at 850 °C. After the oven is cooled down, the black mixture is removed into the Mortar and ground with addition of acetone, for 30 minutes. Again, the mixed gray powder is placed into the oven for 5 hours at 850 °C. As final step, oven is cooled down to the room temperature, and the hard burned powder is ground for 30 minutes and taken to the analysis afterward.

Following the same above way, sample 2 and 3 were prepared with 90%, 80% in Sequence.

### 3.4 X-RAY measurements

The x-ray diffractometer used is D8 focus which is triple-axis x-ray diffractometer that consists of a goniometer instrument to give  $(2\theta)$  and out fitted with a Cu-x-ray tube with two beam slits, with a germanium detector, the tube has a filter wheel to remove the  $(k_{\beta})$  radiation, while allow  $(k_{\alpha})$  radiation to pass through.

The sample is mounted on 1 inch glass disc for the diffraction run and spun in the plane of the glass disc to increase the random orientation of powder grains in the mount.

The pulse height analyzer is used to process the detected x-ray pulse from the germanium detector that set for narrower discrimination of Cu  $k_{\alpha}$  pulse and reject other energy ranges.

The acquisition conditions are 40 KV and 40 mA and scans are obtained typically from 20-80 degrees of 20 with step size of 0.02 degrees and 1 count at constant time, every 5 seconds. Raw diffraction scans which stripped of  $k_{\alpha 2}$  component background corrected with a digital filter and the peaks are investigated using variety of algorithms.

## Photos of the samples during preparations







# CHAPTER FOUR RESULTS AND DISCUSSION

## 4.1 Results

The three samples of YBCO with different Yttrium Oxide concentration are characterized via XRD and the results are given below for the main structural properties

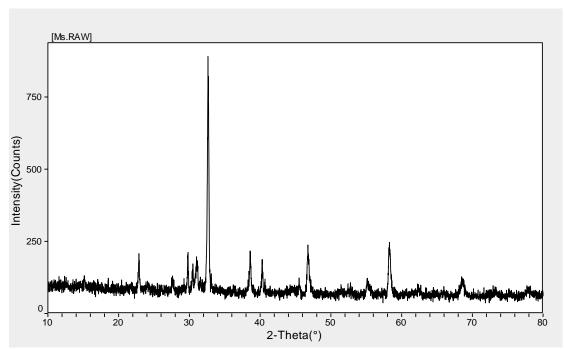


Fig (4.1) XRD spectrum of YBa2C3O7 – 100% sample

Table (4.1) Calculate Lattice Constants from Peak Locations and Miller Indices [Orthorhombic – Primitive] of YBa2C3O7 sample (1)

Table (4.1) Lattice Constants from Peak Locations and Miller Indices

20	d ( nm )	h k l
12.367	7.1512	1 0 1
22.865	3.8861	1 0 0
24.071	3.694	0 0 1

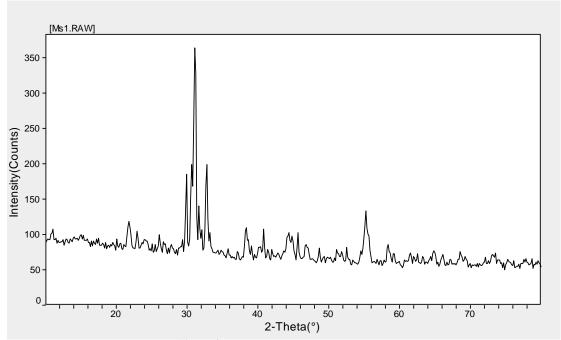
Average Lattice Constants = 4.4318

a= 3.8807 b = 11.7355 c = 3.8336 / 
$$\alpha = \beta = \gamma = 90^{\circ}$$
, Density =

6.2689 mg.cm-3

Crystal Form: Orthorhombic – Primitive

Space Group: P m m m (47)



Cell Volume:  $(174.6 \text{ x} 10^{10} \text{ m})^3$ 

Fig (4.2) XRD spectrum of  $YBa_2C_3O_7 - 90\%$  sample

Table (4.2) Calculate Lattice Constants from Peak Locations and Miller Indices [Tetragonal – Primitive] of YBa2C3O7 sample (2)

Table (4.2) Lattice Constants from Peak Locations and Miller Indices

20	d ( nm )	h k 1
17.556	5.0476	0 0 2
20.531	4.3223	2 0 0
21.531	4.1010	1 0 1

Average Lattice Constants = 4.4318

a= 9.138 b = 8.138 c = 8.096 / 
$$\alpha = \beta = \gamma = 900$$
 Density =

5.2576 mg.cm-3

Crystal Form: Tetragonal – Primitive

Space Group: P m m m(123)

Cell Volume: (536.17 x1010 m)3

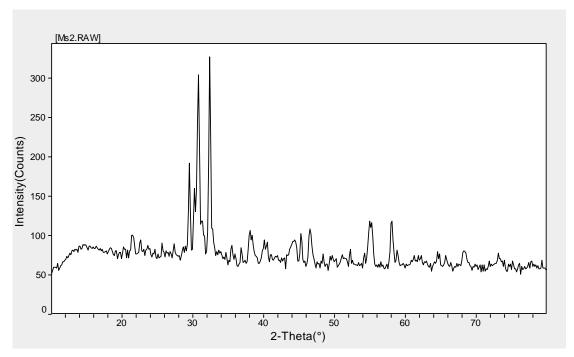


Fig (4.3) XRD spectrum of  $YBa_2C_3O_7 - 80\%$  sample

Table (4.3) Calculate Lattice Constants from Peak Locations and Miller Indices [Orthorhombic – A- Center] of YBa2C3O7 sample (3)

Table (4.3) Lattice Constants from Peak Locations and Miller Indices

20	d (nm)	h k l
29.517	3.0237	1 0 0
30.264	2.9508	1 0 5
32.311	2.7684	0 1 13

Average Lattice Constants = 4.3152

a = 3.842 b = 3.881 c = 50.1 /  $\alpha = \beta = \gamma = 900$  Density = 3.1105

mg.cm-3

Crystal Form : Orthorhombic – A-Center

Space Group: A m m m(65) Cell Volume: (753 x1010 m)3

#### 4.2 Discussion

In this research, the yttrium barium copper oxide (YBCO) compound was prepared as one of the compounds with superconductivity properties at temperatures of about 93 degrees Kelvin. Studies have shown the possibilities of developing this compound to reach superconductivity at temperatures close to room temperature.

When studying the prepared samples in the way of X-ray diffraction (XRD), it was found that the samples correspond to the reference sample in determining the peak of the diagram, which indicates that the samples prepared for the compound are good and give a superconductor in initially. This can be confirmed by conducting a study on the various applications for which the possibilities are not available and will be recommended in the recommendations.

It was found that the three prepared samples differ in their structural properties in terms of the size and shape of the primitive unit cell due to the change of the concentration of yttrium oxide in the compound, which is the main element, and this leads to the possibility of further experiments to reach the ratio of yttrium oxide in the compound that gives the best superconductive properties for this compound.

The first sample was of 100% yttrium oxide and gave results for the primitive unit cell and Miller indices as follows: Average Lattice Constants = 4.4318

a= 3.8807 b = 11.7355 c = 3.8336 / 
$$\alpha = \beta = \gamma = 90^{\circ}$$
 Density = 6.2689 mg.cm-3

Crystal Form: Orthorhombic – Primitive

Space Group: P m m m(47)

Cell Volume: (174.6 x1010 m)3

These are considered to be more than 98% identical with reference to the reference values referred to in Chapter 2. When the yttrium oxide was changed to 90%, the results were as follows: Average Lattice Constants =

4.4318 a= 9.138 b = 8.138 c = 8.096 / 
$$\alpha = \beta = \gamma$$

 $= 90^{\circ}$  Density = 5.2576 mg.cm-3

Crystal Form : Tetragonal – Primitive

Space Group: P m m m(123)

Cell Volume: (536.17 x1010 m) 3

Which is also indicates that the properties are identical with the reference values with a difference in the shape of the primitive unit cell and the dimensions of its primitive unit cell.

In the third sample of 80% of yttrium oxide, the cell returned to the first form with a slight difference in the dimensions of the unit cell and the Miller indices.

All of the above results clearly indicate the success of the preparation of the compound and give good results with weak potential to conduct further tests on the samples to give a clearer picture in confirming the superconducting property of this compound at specific temperatures and find the best possible degree, and conduct various applications on the compound such as Joseph Son's connection and levitation phenomenon.

### 4.3 Conclusion

Three sample of YBCO alloy were prepared .In the first sample where Y2 O3 ratio was 100% the structure was orthorhombic, In the second where Y2 O3 ratio was 80% the structure was tetragonal . In the third sample where Y2 O3 was 80% the orthorhombic structure appeared again with a difference in space group and values of a, b and c.

### 4.5 Recommendations

It is recommended that further tests should be carried out on the YBCO compound such as the Scan Electro Microscope (SEM) test of the Tc measurement and conducting and testing on applications such as levitation.

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