



## Effect of the Structural and Electrical Properties of $\text{Bi}_{2-x}\text{Pb}_x\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ Superconductors with Partial Substitution of Lead by Bismuth

**Alyaa H. Ail Jassim**

Department of Physics, College of Education for Pure Sciences, University of Kirkuk, Iraq.

\*Corresponding Author: [alyaahamid@uokirkuk.edu.iq](mailto:alyaahamid@uokirkuk.edu.iq)

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### Abstract:

This manuscript discusses the preparation of  $\text{Bi}_{2-x}\text{Pb}_x\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  compounds by the method of solid-state reaction at the annealing temperature  $850\text{ }^\circ\text{C}$ . Under pressure of  $8\text{ ton/cm}^2$  with a presence of enough oxygen and these are considered as ideal conditions according to the previous researches in the preparation of electric superconductors of high degree temperatures the effect of partial substitution was on Lead (Pb) element in the Bismuth (Bi) element was studied to produce a compound with formula of  $\text{Bi}_{2-x}\text{Pb}_x\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  and for different ratios of x to know the effect of partial substitution to form Bi2223 phase at the annealing time 72 hrs and we concluded from study of diffraction of x-ray that the compound kept its tetragonal structure, and critical temperature ( $T_c$ ) degrees were obtained  $T_c = 128\text{K}, 130\text{K}, 135\text{K}, 132\text{K}$ , and that's at substitution ratios  $x=0, 0.15, 0.25, 0.3$ , respectively it was clear the best substitution ratios of pb in Bi was when  $x=0.25$ .

**Keywords:** Bismuth, Lead, Superconducting Properties, Solid State Reaction Method, Tetragonal Structure.

## تأثير الخواص التركيبية والكهربائية لموصلات $Bi_{2-x}Pb_xBa_2Ca_2Cu_3O_{10+\delta}$ الفائقة عند الاستبدال الجزئي للرصاص بالبرزموت

عليا حامد علي جاسم

قسم الفيزياء، كلية التربية للعلوم الصرفة، جامعة كركوك، العراق

[alyaahamid@uokirkuk.edu.iq](mailto:alyaahamid@uokirkuk.edu.iq)

### الخلاصة:

يدرس هذا البحث إعداد مركبات  $Bi_{2-x}Pb_xBa_2Ca_2Cu_3O_{10+\delta}$  باستخدام طريقة التفاعل في الحالة الصلبة عند درجة حرارة تليد تبلغ 850 درجة مئوية، وتحت ضغط 8 طن/سم<sup>2</sup>، مع وجود كمية كافية من الأوكسجين، وتعتبر هذه الظروف مثالية بناءً على الأبحاث السابقة في تحضير الموصلات الفائقة الكهربائية ذات درجات الحرارة العالية. تمت دراسة تأثير الاستبدال الجزئي لعنصر Pb بعنصر Bi لإنتاج مركب بصيغة  $Bi_{2-x}Pb_xBa_2Ca_2Cu_3O_{10+\delta}$  و لتراكيز مختلفة لـ x، وذلك لمعرفة تأثير الاستبدال الجزئي على تكوين الطور Bi2223 عند زمن تليد بلغ 72 ساعة. ومن خلال دراسة حيود الأشعة السينية (XRD)، تبين أن المركب يحافظ على صفته الرباعية (tetragonal)، وتم قياس درجات الحرارة الحرجة حيث كانت القيم 128K، 130K، 135K، 132K عند نسب استبدال  $x = 0, 0.15, 0.25, 0.3$  على التوالي. وقد كان واضحاً أن أفضل نسبة استبدال لعنصر Pb بعنصر Bi هي عند  $x = 0.25$ .

**الكلمات المفتاحية:** البرزموت، الرصاص، خصائص الموصلية الفائقة، طريقة التفاعل في الحالة الصلبة، التركيب الرباعي.

### 1. Introduction:

When materials are cooled to a specific temperature, known as the  $T_c$ , they become superconducting, allowing electric current to flow without resistance and with no magnetic flux at all. This property is crucial for many applications in electronics, connectivity, and medical equipment. [1,2]. After the discovery of some ceramic materials with a temperature of more than 90 K, which were referred to as high-temperature superconductors, superconductors entered a new stage. The ability to use liquid nitrogen for cooling [3] and liquid nitrogen at a temperature of (77 K), which can be obtained readily and at an affordable price [4,5], and the discovery of these materials have mad.

High temperature superconducting materials, including the BBCCO system, have been identified and synthesize [6] is an abbreviation for HTSC, composed of element oxides (bismuth, barium, calcium, copper) with the typical chemical formula  $(Bi_{2-x}Pb_xBa_2Ca_{n-1}Cu_nO_{6+\delta})$ , where  $(n = 1, 2, 3)$ . It is of significant significance because of its high frequency, elevated temperature tolerance, and excellent chemical resistance to moisture. Consequently, significant efforts have been devoted to examining the preparation methods, treatments, and

properties of this system [7,8]. The superconducting system (BBCCO) exhibits a layered structure including copper oxide layers (CuO) with Critical temperatures ( $T_c$ ) of 110, 80 and 10 K, respectively, at which the electrical resistance reaches zero ( $R=0$ ) [9,10]. The superconducting and transition properties of high-temperature CuO compounds are very different [11,12]. The unsupported alternating conductivity at frequency, coupled with a low insulation constant at high frequencies (60 GHz) at room temperature, and the material's polarization may necessitate the use of frequency-dependent conductivity [12].

The discovery of high temperature superconducting material initiated a revolutionary advancement in industrial applications and materials science [13,14]. High temperature superconducting systems include Bi-2201, Bi-2212, and Bi-2223 [15], which are distinguished by their two-dimensional basis and 1-layered structure, comprising three phases: Bi-2201, Bi-2212, and Bi-2223. A final numeral for each phase denotes the quantity of CuO layers, which corresponds to the  $T_c$  (10K, 80K, and 110K); the latter signifies the temperature at which electrical resistance is zero ( $R = 0$ ) [16]. The Bi-2223 phase is challenging to synthesize, although it possesses the advantage of being a single phase with the highest  $T_c$  (110 K) among 204 effects of partial substitution of silver and copper on the structural and electrical characteristics of  $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  superconductors across three phases [17]. The characteristics of superconductors can be adjusted by incorporating or eliminating an element with varying ionic radius and bonding properties, and the enhancement or decline in superconducting properties is contingent upon the attributes of the added or substituted elements that differ in radius and bonding. The majority of the research concentrates on enhancing the morphology and characteristics of (Bi-2223) through substitution studies [18-20]. This current work discusses the temperature-dependent electrical characteristics and structural properties of the  $\text{Bi}_{2-x}\text{Pb}_x\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  samples and at concentrations  $x=0, 0.15, 0.25$  and  $0.3$ .

## 2. Material and methods:

The molecular weights of these materials relative to the weight of the element in each of the base material and the compound (sample) to be generated were used to calculate the weight ratios of the materials that contribute to the creation of the  $\text{Bi}_{2-x}\text{Pb}_x\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  compound.

The oxides and carbonates are weighed out, combined, and then put into granules. These materials are then ground finely for 30 minutes using an agate grinder so that the mixture becomes homogeneous. An isopropanol solution is added during the grinding process to prevent falling or losing powder during the grinding process. The isopropanol alcohol is then removed from the grinder by placing it in an electric oven set between ( $50^\circ\text{C}$ ,  $60^\circ\text{C}$ ).

These powders are put in a convection oven, which is heated to 850°C and heated at a pace of 120°C per hour in an air-saturated environment. This form is kept at (850 C) for (12) hours before being lowered to allow it to cool to a specific temperature. The model is taken out of the furnace at room temperature with a cooling rate of 30°C/hr and temperature control using a thermocouple. To prevent vaporization and loss, the powder is then combined and continuously processed for another 30 minutes. The ester of is solution is then added. 50°C, 60°C).

The powder is then manufactured under pressure (8 Ton/cm) in the form of tablets. These tablets had (12 mm) diameters and ranged in thickness from (0.8 mm) to (1.2 mm). These discs are put in an electric oven, where the temperature is progressively increased to (850 C°) at a pace of (120 C° /hr) and held there for 12 hours before being gradually lowered at a rate of (30 C°). After acquiring the samples that were made in the shape of tablets from the previous sentence, the process of heating and cooling takes place in an atmosphere that is saturated with oxygen until it reaches room temperature. This process is known as sintering. The prepared samples are obtained as tablets, which are then placed in an electric oven and heated to (600 C°) at a rate of (120 C° /hr) from room temperature. The sample is then kept at this temperature for (12) hours before the temperature is raised. The oven heats up to 850 degrees Celsius at a rate of 120 degrees Celsius per hour, where it stays for 24 hours in an oxygen-rich environment. The model then cools to 600 degrees Celsius at a rate of 30 degrees Celsius per hour, where it stays for another 12 hours before being cooled to room temperature at a rate of 60 degrees Celsius per hour to room temperature. Tablet-shaped samples are obtained, and after being processed, they will be examined using an X-ray diffraction instrument to determine their structural characteristics and to look at their electrical properties with the four probes method.

### 3. Results:

When the element pb is partially substituted for the element Bi in the compound  $\text{Bi}_{2-x}\text{Pb}_x\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  compound, with  $x=0, 0.15, 0.25, \dots$ , the structural properties of the compound have been studied. However, the structural properties of the compound  $\text{Bi}_{2-x}\text{Pb}_x\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ , which was prepared at an annealing temperature of 850 and under a hydrostatic pressure of 8 tons/cm<sup>2</sup> are different. When creating the models with the X-ray device and putting the value of x into the compound, the X-ray diffraction investigation of these samples revealed. The regularity of the crystalline structure and the appearance of distinct peaks are noticed as shown in **Figure 1**.

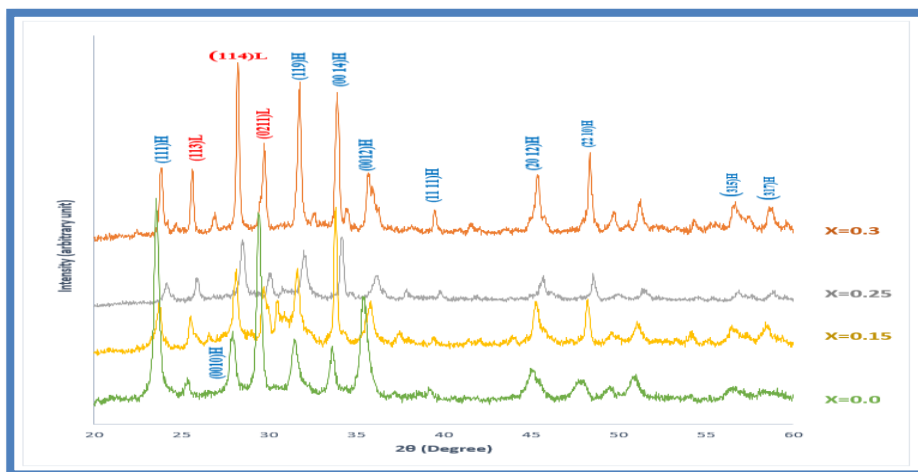


Figure 1. X-ray diffraction of  $\text{Bi}_{2-x}\text{Pb}_x\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  compound as function of  $2\theta$ , with indicated values of  $x=0,0.15,0.25, x=0.3$

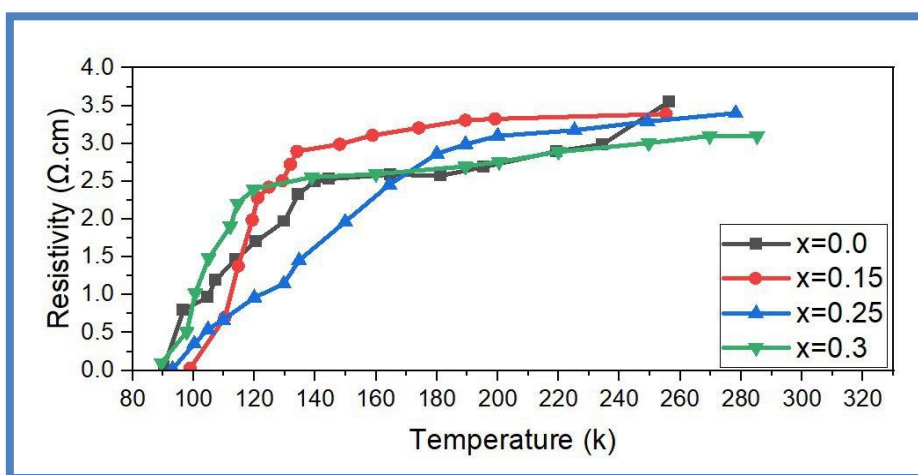


Figure 2: Resistivity of  $\text{Bi}_{2-x}\text{Pb}_x\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  compound as function of  $\text{pb}$ , with indicated values of  $x=0,0.15,0.25, x=0.3$

Figure 2 presents the electrical resistance behavior as a function of temperature for the four samples, both before and during the partial replacement. The maximum zero-resistivity ( $T_c$ ) of 135 K was achieved at  $x = 0.25$ ; however, at other concentrations,  $T_c$  increased with  $x$  concentration, as shown in Table 1.

Table (1): Values axes  $a$ ,  $c$ ,  $T_c(\text{onset})$ ,  $\Delta T$  and  $T_c(\text{offset})$  of  $\text{Bi}_{2-x}\text{Pb}_x\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  compound as function of  $\text{pb}$ , with indicated values of  $x=0,0.15,0.25,0.3$

X	$a$ (°Å)	$c$ (°Å)	$c/a$	$T_c$ (OFF)(K)	$T_c$ (ON) (K)	$T_c$ (K)
0	5.0923	35.017	6.97	95	150	123
0.15	5.339	36.895	6.91	198	160	129
0.25	5.432	37.028	6.81	100	170	135
0.3	4.973	36.962	7.64	99	122	110

#### 4. Discussion

The regularity of the crystalline structure and the appearance of distinct peaks are noticed as shown in **Figure 1**.

Miller coefficients hkl are then discovered, and using a particular BASIC software, the values of the unit cell's dimensions are discovered.  $a = b = 5.09 \text{ \AA}$  and,  $c = 35.01 \text{ \AA}$  for  $x = 0$ , where:  $a = b = 5.33 \text{ \AA}$ ,  $c = 36.89 \text{ \AA}$  for  $x = 0.15$ ,  $a = b = 5.43 \text{ \AA}$  and,  $c = 37.02 \text{ \AA}$  for  $x = 0.25$ , as shown in **Table 1**. The table indicates that elevating the concentration of partially transcendent elements results in a significant alteration of both the network constants and their size. An increase in the value of  $c$  resulted in variations in the concentrations  $x = 0.0, 0.15, 0.25, 0.3$ , attributed to disturbances in the number of CuO chains and CuO<sub>2</sub> levels that govern the various isotropic materials and the  $T_c$  of superconducting materials. A decrease in the coefficient  $c$  diminishes the intensity of the fermi energy levels, thereby lowering the  $T_c$ , causing imbalance and variability, and affecting the unit cell volume in a compound [21]. The change in lattice constants arises from the variation in ionic radii of the substituted elements resulting from an increase in substitution concentration. X-ray diffraction measurements indicated that the crystalline structure remained tetragonal [22], with a noticeable reduction in the  $c$ -axis length when the compensation ratio increased to  $x = 0.3$ , attributed to the relocation of atomic defects or oxygen deficiency. The vacuoles or abnormalities of the positive ions result in the accumulation of stacking faults. The distortion of the crystal structure eventually results from the influence along the ( $c$ ) axis [23].

As shown in **Figure 2** the results indicate that all samples exhibited metallic behavior, with the  $T_c$  rising in accordance with the increasing  $x$  concentration. The electrical resistance decreases as the temperature decreases in the region preceding ( $T_c(\text{onset})$ ), as the material transitions from its natural state. The change to the superconducting state occurred in several stages, influenced by several transitions arising from the various phases present in the sample, as well as the presence of certain crystals and impurities. The findings indicated that the  $T_c$  varies with increasing  $x$  concentration [22]. The maximum zero-resistivity ( $T_c$ ) of 135 K was achieved at  $x = 0.25$ ; however, at other concentrations,  $T_c$  increased with  $x$  concentration, as shown in **Table 1**.

This result can be explained by the compound playing the perfect role in the crystal structure [20], and this percentage of compensation caused the  $T_c$  to rise as a result of an increase in the high phase 2223 with an increase in the lead concentration in the samples.

Using the four-probe approach, the electrical characteristics of the compound were investigated in order to determine  $T_c(\text{onset})$  and  $T_c(\text{offset})$  for calculating the electrical resistivity as a function of temperature after the element pb was partially substituted in the Bi element of the  $\text{Bi}_{2-x}\text{Pb}_x\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  compound.

## 5. Conclusions

The effect of partial bismuth replacement with lead on the superconducting characteristics of  $\text{Bi}_{2-x}\text{Pb}_x\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  is examined in this publication with  $x = 0, 0.15, 0.25$  and  $0.3$ . The solid-state reaction approach was used to prepare the samples. The four-probe method was used to evaluate the electrical conductivity in order to calculate the  $T_c$ . The  $T_c$  was observed to rise as the quantities of silver in all produced samples increased. The optimal compensation ratio for  $x$  was also discovered to be  $0.25$ , which results in the highest  $T_c$ . X-ray diffraction was used to examine the samples' ultrastructural characteristics. All of the prepared samples had a tetragonal structure, with a distinct change in the lattice constants, according to an X-ray diffraction investigation. The ideal role that the compound played in the crystal structure can be utilized to explain this result. This percentage of compensation led to an increase in the  $T_c$  and an increase in the high phase 2223 with an increase in the lead concentration in the samples.

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