

## Structural and electrical properties of the high temperature superconducting system



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### الخلاصة

في هذا البحث حضرت النماذج ( $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ ) على شكل أقراص صلبة وفي حالات مختلفة من عمليات التلييد وبدرجات حرارة مختلفة (810, 830, 850, 870, 890). استخدمت تقنية الفحص بالأشعة السينية (XRD) لمعرفة التركيب البلوري للمركب ومن خلال الفحص تم معرفة ان الحصول على تركيب تتراكونال مع معلمات الشبكة ( $a=b=5.42\text{Å}$ ,  $c=34.13\text{Å}$ ) عند درجات حرارة تلييد (850) م° وان المقاومة الكهربائية للنماذج ال ملبدة عند (850) م° لها درجة حرارة حرجة مقدارها ( $T_c=120\text{K}$ ) حيث قلت هذه الدرجة الى (87K) عند درجة حرارة تلييد مقدارها (870) م°. وعندما تم تلييد نفس النماذج بدرجة حرارة (890) م° أظهرت صفات أشباه الموصلات وبطاقتي تنشيط (3.456-8.593) ملي الكترون فولت . أما تأثير التلدين على درجة الحرارة الحرجة فقد ظهر واضحاً على النماذج المحضرة في درجة حرارة تلييد (850) م°. ان أفضل قيمة لدرجة الحرارة الحرجة  $T_c$  هو (120K) عند درجة حرارة تلييد (400) م° وترتفع هذه الدرجة الى (140K) بزيادة وقت التلدين من (٢٤) ساعة الى (٧٢) ويعزى ذلك الى زيادة نسبة الأوكسجين ونمو طبقة (Cu-O) في الخلية.

### Abstract

Samples of the high temperature superconducting system,  $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ , were prepared by solid state reaction method with different sintering temperatures (810, 830, 850, 870, 890) C°. X-ray

diffraction technique was used to examine the structure of the compound and showed that the sample prepared with sintering temperature at 850 C° exhibits tetragonal phase with the lattice parameters  $a = b = 5.42 \text{ \AA}$ ,  $c = 34.13 \text{ \AA}$ . The electrical resistivity measurements exhibited that the sample prepared at 850 C° its critical temperature was  $T_c = 120\text{K}$  and decreases to 87K when prepared at 870C°. However when the same sample prepared at 890 C° it showed semiconducting behavior with two activation energies (8.593 - 3.456)meV. The effect of annealing on critical temperature has been investigated on the sample which was prepared at 850 C°. The best value of  $T_c$  was (132)K at the annealing temperature 400 C°, and rises to 140K with increasing the annealing time from 24 hrs to 72hrs and this increase might be caused by the increase of oxygen content or by intergrowths of a large number Cu-O layers in the cell.

**Keywords:** High temperature superconductors, semiconductors, electrical resistivity, annealing temperature.

### Introduction:

The discovery of superconductivity between 7K and 22K in the Bi-Sr-Cu-O compound has been reported by Michel et al in 1987 [1]. Because of the intense interest in the 90K material at that time, their report did not attract widespread interest. However, attention quickly was focused on the Bi-containing superconductor in January 1988, when Meada et al [2] and Chu et al [3] reported that adding Ca to the Bi-Sr-Cu-O system produced material that was superconducting above liquid nitrogen temperature 77K. Since then great attention was focused on the series compound Bi-Sr-Cu-O which shows superconductivity around 105K [4]. The properties of these systems show that superconductors with structural formula  $\text{Bi}_2\text{Sr}_2\text{CuO}_6$ ,  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$  and  $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$  have  $T_c$  values of 10K, 85K, and 110 K respectively[5].

The aim of this paper is to investigate the structural and electrical properties of the superconducting compound Bi-Ba-Ca-Cu-O prepared using a special preparation method. In this method, the compound was annealed at several temperatures and for different annealing times to study the effect of preparation conditions on the structural and electrical properties of the superconducting system.

### Experimental:

High temperature  $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$  superconductor samples were prepared using the solid state reaction method. The appropriate quantities of highly pure  $\text{Bi}_2\text{O}_3$ ,  $\text{CaCO}_3$ ,  $\text{BaCO}_3$  and  $\text{CuO}$  have been determined in stoichiometric ratio of Bi:Ba:Ca:Cu =2:2:2:3.

The mixture was ground with isopropanol by using an agate mortar. The well mixed powder of these oxides was calcimined at (800)C°. The mixture was pressed into approximately 1gm pellets, with 1.1cm diameter and 0.17cm thickness. The pressed pellets were initially heated in air from room temperatures to different sintering temperatures (810, 830, 850, 870, 890)C° for 24hr and then slowly cooled (30 C°/hr) to room temperature. Finally, the sample pellets were annealed at different temperatures (100, 200, 300, 400, 500, 600)C°, and for different times (24, 48, 72, 96) hrs.

X-ray diffraction patterns of these samples at room temperature were obtained using Phillips X-ray diffractometer with CuK $\alpha$  source and 1.5418A° wavelength. The electrical resistivity method has been used to determined the critical temperature. The electrical resistivity was measured using the standard four-probe method. Electrical contacts were made using fine copper wires and conductive silver paste. The oxygen content in the sample was measured by using iodometric method[6].

### **Results and Discussion:**

X-ray diffraction patterns of Bi<sub>2</sub>Ba<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+ $\delta$</sub> . Fig(1) show the existence of single tetragonal phase for the sample that was prepared at 810 C° as shown in Fig(1). A computer program was used to calculate the values of the lattice parameters a, b and c. Table (I) shows the values of these parameters. Similar behavior has been shown by the sample which was prepared at 850 C°. This was attributed to the change in the structure from orthorhombic to tetragonal with lattice parameters a=b=5.42 A° and c=34.13 A°[7,8,9]

The change in the structure from tetragonal to orthorhombic phase occurred also in the samples which were prepared at 870 C° as shown in table I. This means that 870 C° was not the ideal sintering temperature. The change in the structure was observed in samples with sintering temperature 890 C°. Similar results were obtained by other authors [10-13] for different compounds. They pointed out that the elongating in the c-axis plays a crucial role in high temperature superconductors. The increase in sintering temperature leads to creation of more oxygen deficiency, which is very important factor for the presence of high temperature superconductivity.

The electrical resistivity at various temperature was measured for all samples and the results are shown in fig.(2). The superconducting transition temperatures T<sub>c</sub> are shown in table II. It is clear from table II that the value of T<sub>c</sub> increases with increasing the sintering temperature, these results are related to the configuration of possible Cu-O planes or chains in the compound.

But the sample which was prepared at 870 C° showed a decreasing in the value of T<sub>c</sub> while the sample which was prepared at 890 C°, showed semiconducting behavior with two activation energies 8.593 and 3.456

meV, as shown in Fig.(3). The first activation energy is due to hopping between localized states, while the second is due to transport to deeper distances in the gap but with less energy. We attribute that to the change in the structure which leads to the change in superconducting behavior. These results are in good agreement with the result for Bi-2223 compounds [14]. The best value of  $T_c$  was 120K for the sample prepared at 850 C°, since these samples were annealed at different annealing temperatures. The best value of  $T_c$  was obtained for annealing temperature of 400 C° for 24hr as shown from Fig (4) and Fig.(5), and it rises from 120K to 132K.

The increasing in  $T_c$  with annealing temperature, (see table III) depends on the increase in oxygen content, which plays the very important role in increasing or decreasing  $T_c$ . The increase might be caused by decreasing of the structural defects and also due to intergrowth of a large number of Cu-O layers in the unit cell. Similar behavior has been detected by Fathi et al [15].

The relation between resistivity and temperature for the sample annealed at 400C° for different times are shown in fig.(5), it is clear that, as the time of annealing is increased the value of  $T_c$  increases and saturates at the annealing time of 72 hr as shown in table IV. The increase in  $T_c$  value with increasing annealing temperature leads to the increase in the charge carriers, the Cooper pairs, which is due to electron-phonon coupling[5].

### **Conclusion:**

Superconductivity is shown to be possible in the tetragonal structure and orthorhombic structure, depending to the sintering temperature. The best value of sintering temperature is 850 C° which gives the best structure and electrical properties. The change in the structure occurs at sintering 890 C° which is due to the change in phase. The best value of  $T_c$  is obtained at annealing temperature 400C° which affects the oxygen content. The value of  $T_c$  increase and saturates at annealing time 72hr. Annealing can cause increase the  $T_c$  it causes the decrease in the structural defects.

**Table I**  
**Lattice parameters for sintering temperature to preparing  $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  superconductors.**

T (C°)	Lattice constant		
	a (A°)	b (A°)	C (A°)
810	5.41	5.41	30.75
830	5.45	5.45	31.03
850	5.42	5.42	34.13
870	5.43	5.40	30.99
890	5.42	5.40	30.88

**Table II**  
**Critical temperatures for examined samples  $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  superconductors with different sintering temperature.**

T (C°)	T <sub>c</sub> (K)	10+δ
810	95	10.12
830	100	10.17
850	120	10.22
870	87	10.71
890	Semiconductors: E <sub>act</sub> (1)=8.593 mev E <sub>act</sub> (2)=3.456 mev	10.27

T = sintering temperature.

T<sub>c</sub>=critical temperature.

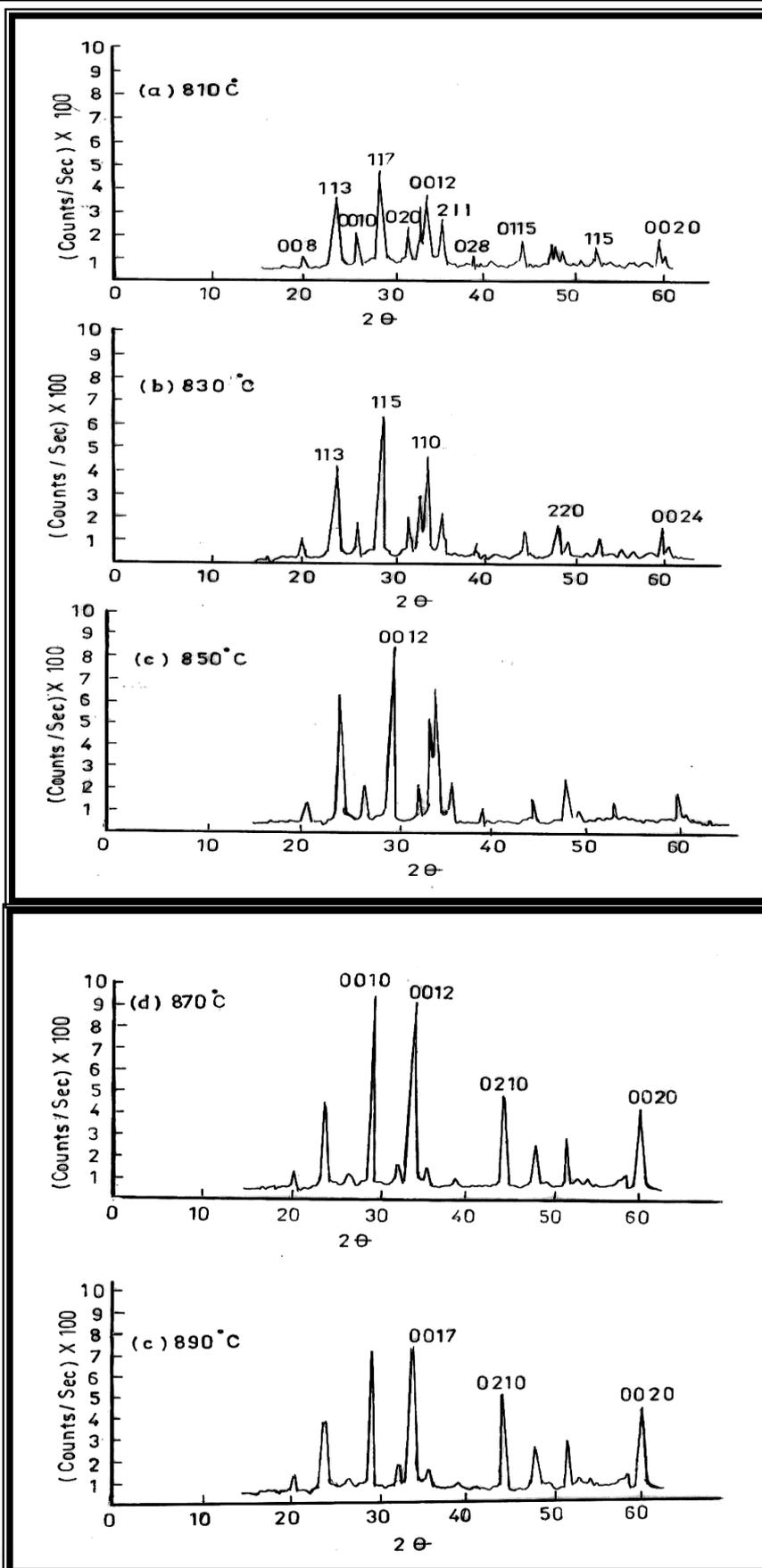
10+δ = Oxygen content.

**Table III**  
**Critical temperatures for annealed  $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  superconductors at 850 C° with different annealing temperature.**

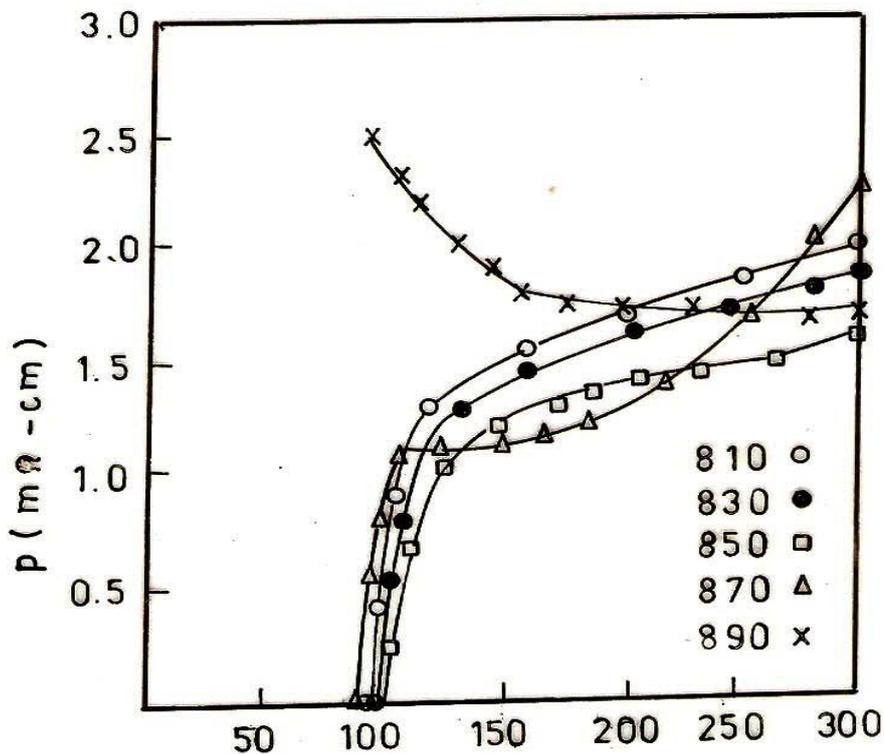
Annealing Temperature T (C°)	Critical Temperature T <sub>c</sub> (K)	10+δ
100	125	10.25
200	127	10.29
300	130	10.30
400	132	10.38
500	90	9.97
600	82	9.93

**Table IV**  
**Critical temperatures for annealed  $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  superconductors at 850 C° with different annealing time for best annealing temperature (400 C°).**

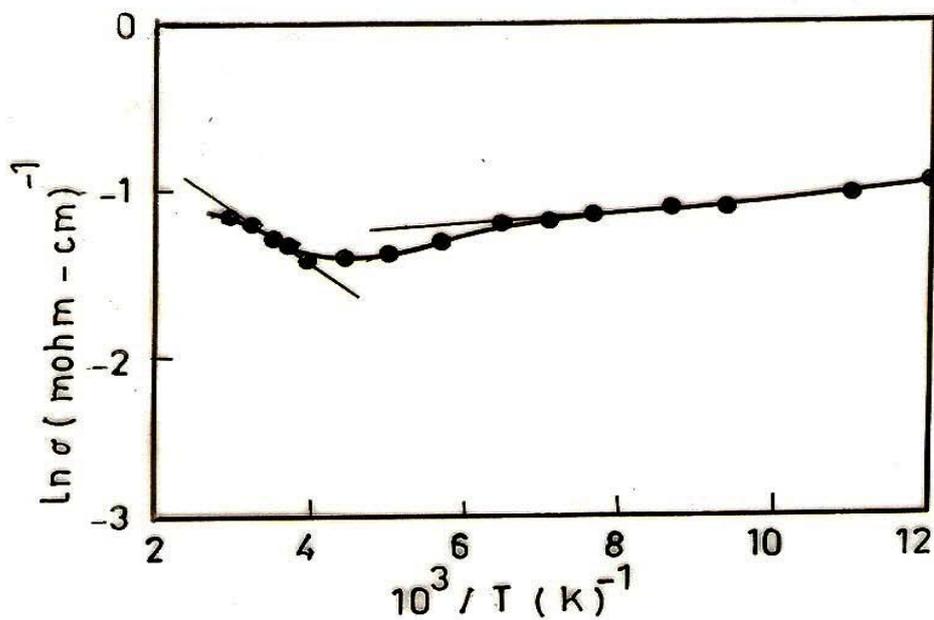
T <sub>c</sub> (K)	Annealing time (hr)	10+δ
132	24	10.38
134	48	10.40
136	72	10.42
136	96	10.42



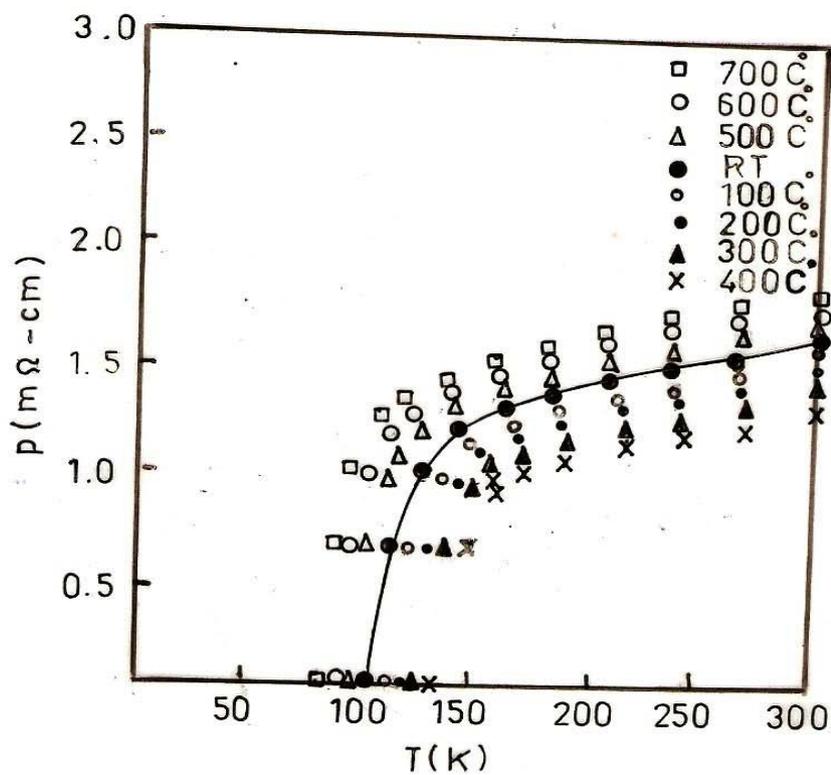
**Fig(1): X-ray diffraction pattern of  $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$  at different sintering temperatures.**



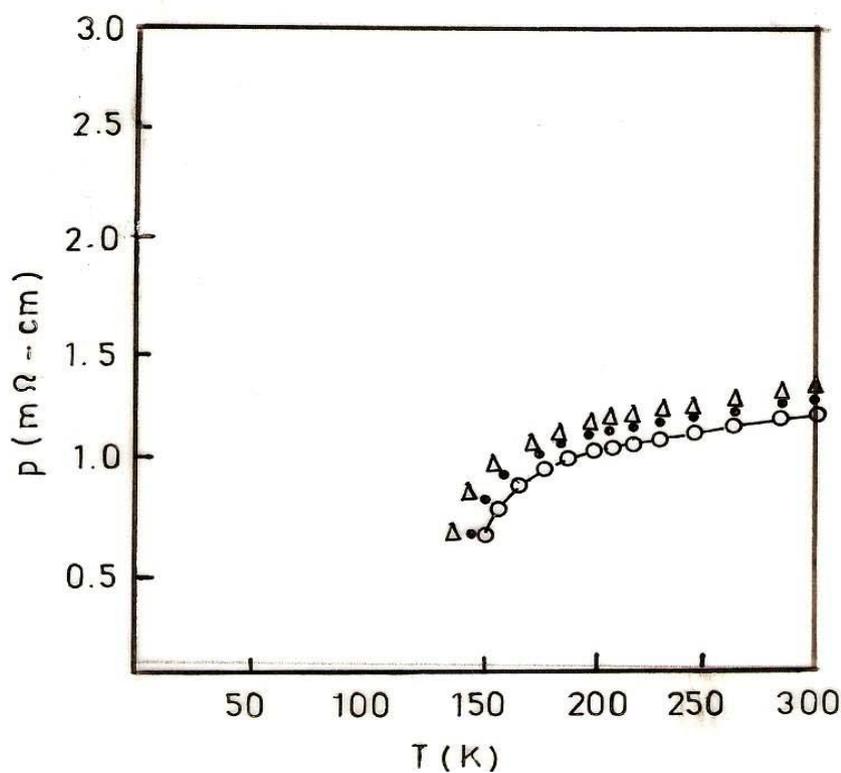
Fig(2): The resistivity versus Temperature  $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$  at different temperatures



Fig(3): The conductivity versus Temperature  $\text{Bi}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$  at 890°C



Fig(4) The resistivity versus Temperature  $Bi_2Ba_2Ca_2Cu_3O_{10}$  at  $850^\circ$  with different annealing temperature.



Fig(5) The resistivity versus Temperature  $Bi_2Ba_2Ca_2Cu_3O_{10}$  at  $850^\circ$  with annealing temperature 4000c at different time.

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