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# Effect of sintering temperature on TC of $Bi_2Ba_2Ca_2Cu_3O_{7-\delta}$ (system 2223)

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## Abstract

samples of high temperature superconductors"$Bi_2$ $Ba_2$ $Ca_2$ $Cu_3$ $O_{7-\delta}$" were prepared by solid state reaction method with different sintering temperature(i.e 790,830,860,890) C°.X-ray analysis techniques were used to examine the structure of the compound, the study showed ,that the sample prepared during sintering temperature at 860 ċ exhibite tetragonal phase with lattice parameter,(a=b=5.408A°,C=30.88A°)while the compound prepared at 890C,° exhibit a change of structure from (tetragonal to orthorhombic) phase this is due to decreasing in(C)axises and increasing in the other two axis's (a,b)and the value of lattice parameters were a=5.418A°,b=5.429A°,C =30.75A°Electric resistively method were used to determine the critical temperature(TC)of these compounds using liquid nitrogen cryostat the compounds prepared at [790,830,860 and 890]C,°· Showed(TC)values of[105, 115,135, and92]K respectively.The change in(TC) values can be explained on the basis that increasing the sintering temperature produces a more uniform crystal structure and an increased oxygen content in the compound.

## Introduction

Superconductivity means that the electrical resistively of many metals, alloys, and recently some ceramic material(Kittle,1976;Kittle,1986)drops suddenly to nearly zero.when the specimens are cooled to a sufficiently low temperature(Turner&Arnorld,1981)this is often occur at a temperature of liquid helium(4.2K)or at liquid nitrogen(77K)for low and Superconducter resectively.The transition,or critical temperature(TC)is The temperature at which the transition from the normal state to Superconductivity state takes place(Kittle,1976)and it is the charateristic of a particular material(Wang,et al.,1988).The phenomenon of Superconductivity has been studied initially by H.Kamerlingh Onnes(Saleh,1996;Grejoy,et al.,1973)in 1911when he observed that the electrical resistively of pure mercury dropped abruptly upon cooling it to(4.2K)(Hatfield,et al.,1988).The discovery of Superconductivity between(7K)and( 22K)in(Bi-Sr-Cu-O)compound has been reported by Michel et al. in 1987,(Al-Jobur&Fathi,1994).Attention quickly focussed on the bismnth containing superconductor in January 1988 when medae, chu and et al(Willis, et al.,1995)reported that adding Ca

to the(Bi-Sr-Cu-O)system produced material that was Superconducting above liquid nitrogen temperature(77K)three Superconducting oxides were subsequently identified in the(Bi-Ca-Sr-Cu-O)(Hazan,et al.,1988)wong and green 1999.

-$Bi_2Ca_2Sr_2CuO6+x$, with(TC=22K), $Bi_2\ Ca_1\ Sr_2\ Cu_2\ O_{8+x}$ with(TC= 85K) and $Bi_2\ Ca_2\ Sr_2\ Cu_3\ O_{10+x}$

With(TC=110 K)for bervity ,these phases will be referred to as Bi 2021, Bi 2122 and Bi 2223 respectively.The pervoskite-like that $YBa_2Cu_3O_7$-x,the Ca and Sr cation in Bi 2122 play the same roles as the Y and Ba cations in $YBa_2Cu_3O_7$-x structure ,the linear chans in $YBa_2Cu_3O_7$-x structure are replaced by the Bi-o bilayers proving that linear chains are not essential for high temperature Superconductivity(Fathi,1993).In this paper,we used the solid state reaction method for preparing the compound($Bi_2Ba_2Ca_2Cu_3O_{7-\delta}$), which is the annealing method for high temperature and different annealing times due to effect on(TC)value and on the oxygen content,observable oxygen content play the major(vital)role in determining the superconductor properties.

## Experimental work

High temperature($Bi_2Ba_2Ca_2Cu_3O_{7-\delta}$)for(2223)system superconductors samples were(useing solid state reaction)and the samples wereprepared from approperiate atomic weight of find powder($BiCO_3$,$BaCO_3$,$CaCO_3$ ,and CuO)all with purity 99% to from the perroskite($Bi_2Ba_2Ca_2Cu_3O_{7-\delta}$) Compounds observable using sensitive balance for account weights,See table(1).The total mass of powder $W_T$was grinded to gether for(40)minutes by using a get mortar with isopropanol($C_3H_8O$)Liquid,then dried for an hour in oven at temperature between(50-60)C°the compound was taken after placing it in a ceramic boat and weight again $G^w$.The powder was sintered with heating rate of(120)C°per hour up to(860)C°,and left at that temperature for twelve hours in air,then slowly cooled down to room temperature with cooling rate(30)C°per hour.The powder of samples was sintered by useing furnaces connected to high temperature controller.The second stage,was weighting the compound again $W_{H1}$,in order to find the loss on mass as follows:-

$$W_1 = W_{G1} + W_{H1} \quad \ldots (1)$$

The compound was again mixed in isopropanol and grind for(40)minutes, then tried from an hour in oven at temperature between(50-60)C°,and weighted again $W_{G2}$.The compound was heated by the same rate of first

stage(i.e.120C°/hr)up to(860)C°and left for(24)hours to remove the rest of($CO_2$)from the compound.The second loss in mass being calaulated as follows:

$$\bar{W}_2 = W_{G2} - W_{H2} \quad ...(2)$$

The total loss in mass during the heat treatment comes to:-

$$W_{loss} = \bar{W}_1 - \bar{W}_2 \quad ...(3)$$

Which should be equal to the mass of the($CO_2$)gas results from the chemical reactions.then,the powder mixed with is operpanol and dried.The fourth stage of the sample preperextion was to press(10 tons\cm²)bout one gram of powder as a pellet with(9 m m)diameter.and((1-1.5)m m)in thickness.The samples were sintered at different temperature as (790,830, 860,890)C°it was found that the best sintering temperature is(860)C°since it give best characteristic.The electrical resistivity(to determination TC) from sintered samples were measured by useing four probe technique,and useing rotary pump at empty volum etorr,using thermocouple in system that size(PTC).$10^{-2}$The critically temperature defined as the temperature at middle point between the resistivity at the onset of the transition and the zero resistivity point.The X-ray diffraction meter which has been used to determine the samples structure has follwing the experimental apparatuse was phillips size.

1-Source=Cuk α Target
2-Range=2000 count/sec
3-Current =20 mA
4-Wave length λ= 1.5418 A°
5-Voltage = $40k_V$
6-Time constant=2 second.
7-Scanning speed=2θ/min.

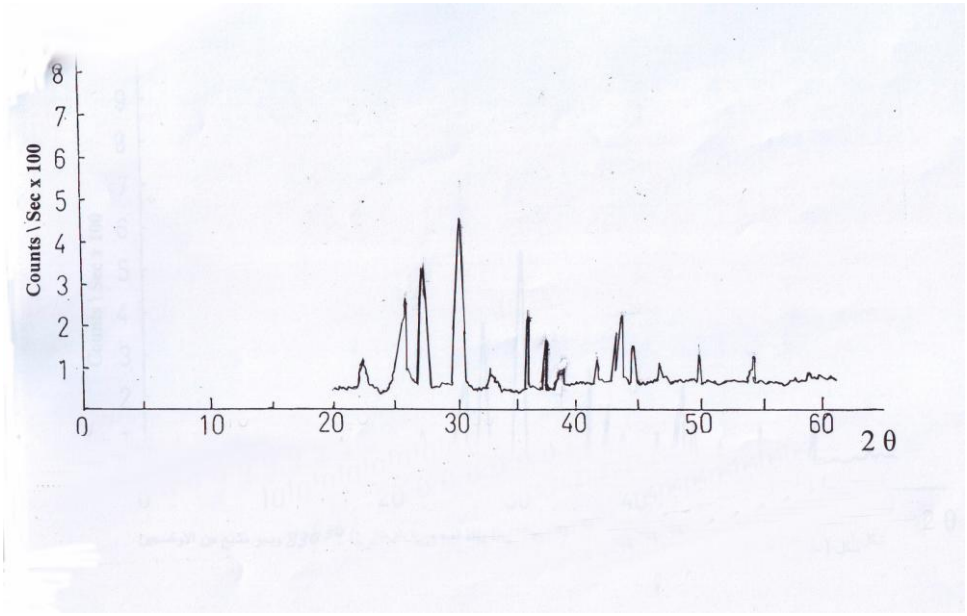
## Results and discussion

X-ray analysis techniques were used to examine the structure for compound($Bi_2Ba_2Ca_2Cu_3O_{7-\delta}$),the study showed the samples prepared during sintering temperature at790 c exhibited prominent apices,wide bases and apices were approaching each other.This is due to the presence of impurities and defect in the samples and indicates that there is homogeneity in the crystal structure as shown in fig(A-1)For the sample sintered at(830) C° the apices were distinct and the bases are less widely which indicates an improvement in the crystal structure as shown in fig(A-2).For the sintering temperature(860C°) the(X-ray),study exhibited high apices, separated from each other, the to

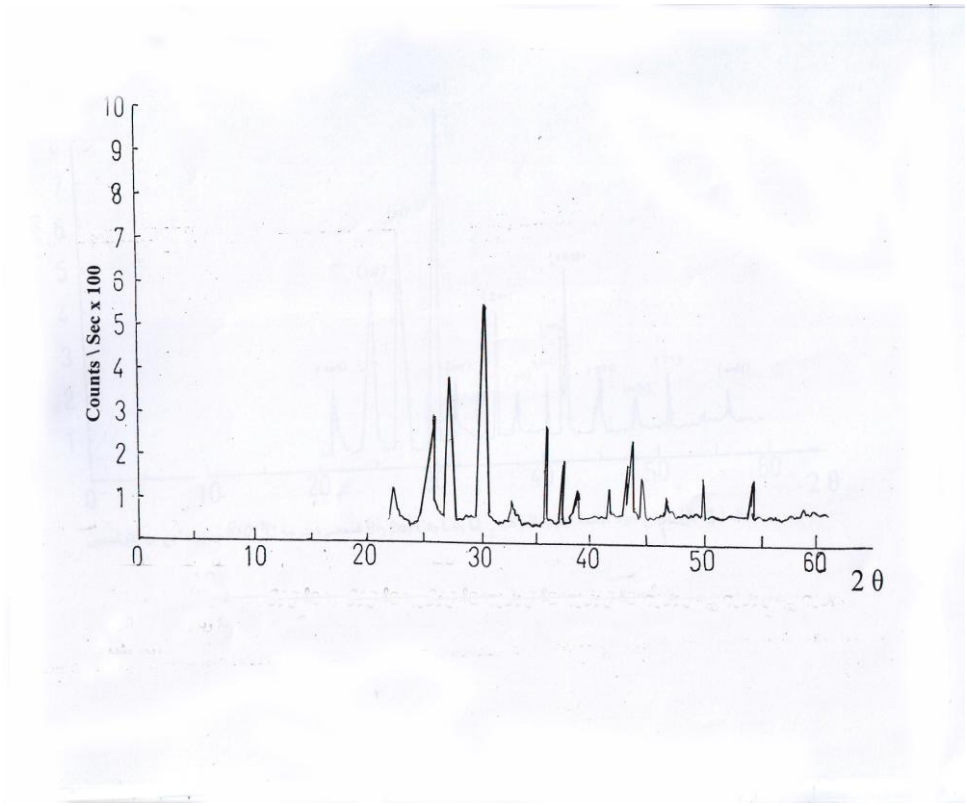
axis's(a,b),exhibited and relaxation which is due to the displacement of cu-planes as shown in fig(A-3).These result are in a good agreement with those reported by(Algbory,1999).This compounds in this state became[Tetragonal] phase ,and the relaxation in(C-axis)is due to an increasing of copper content in the compounds and the effect of sintering temperature and,also an increasing of oxygen content in the structure,all these lead to ordering in the structure,as shown in table(2)which shows the reflection angle.Finally in the sintering temperature(890)C$^{\circ}$it seems that there is a change in the structure of compound,it is noted that C-axis increase and the phase of compound changes such as that–[Tetragonal to Orthorhombic]due to the melting,as show in fig(A-4)This result is a good agreement with(Fathi,1993).We note high apices which are separated from one another and it an effects on the separation of planed from one another and a preanence of small apices(CuO)the transition can be explained to provided Oxygen content,this is be have benefit to repet frost between cationing As show in table(3)which shows the reflection angle. Electrical resistively method has been used to determine critical temperature (TC)of these compound using liquid nitrogen the measured(TC) values for the samples prepared in this study range from(92K-to-135K). depending on the sintering temperature.the samples sintered at(790C$^{\circ}$)exhibit, as(TC)value of (105K)as shown in fig(B-1),this is due to the presence of defects and irregularities in the structure.the sample sintered at(830)C$^{\circ}$exhibit as(TC)value of(115K),the is probably due to an increase in the oxygen content of these samples and also due to an increase in the value of(C-axis)of the structure(B-2),the sample sintered at(860)C$^{\circ}$showed as(TC)value of(135K),this is attributed to a higher regularity in the structure an increase in C-axis value where the structure of the compound become(Tetragonal),and also probably due to the presence of more free paths for copper pairs as shown in fig(B-2). For samples sintered at890 the value of(TC)in(92K)this could be due to the start of melting of the sample near this temperature and the presence of (CuO) impurities in the compound as shown in fig(B-1)table(4)(Fathi,etal,2005).

## Conclusion

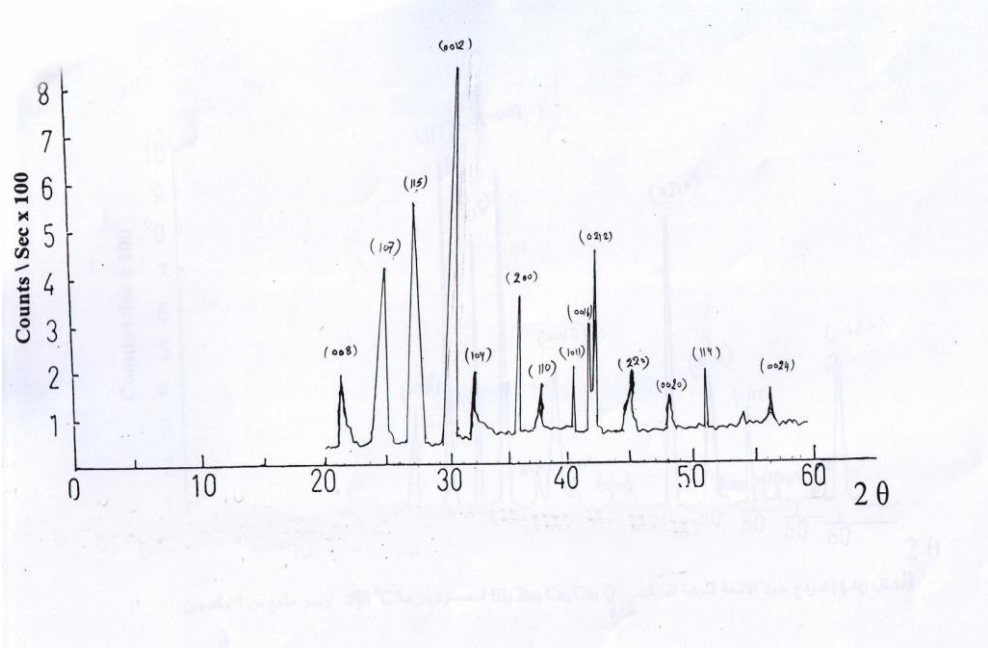
The best value of(TC)is obtained for($Bi_2Ba_2Ca_2Cu_3O_{7-\delta}$)prepared at(860) C$^{\circ}$The determine a value of TC for this case is(135K)this improved value of(TC)can be attributed to an increase of oxygen content.For sample repared at(890)C$^{\circ}$,the value of(TC)decreases,the reason could be a change of phase the structure upon sintering temperature(900)C$^{\circ}$,the sample behaved as non-superconductor is due to solution sample.



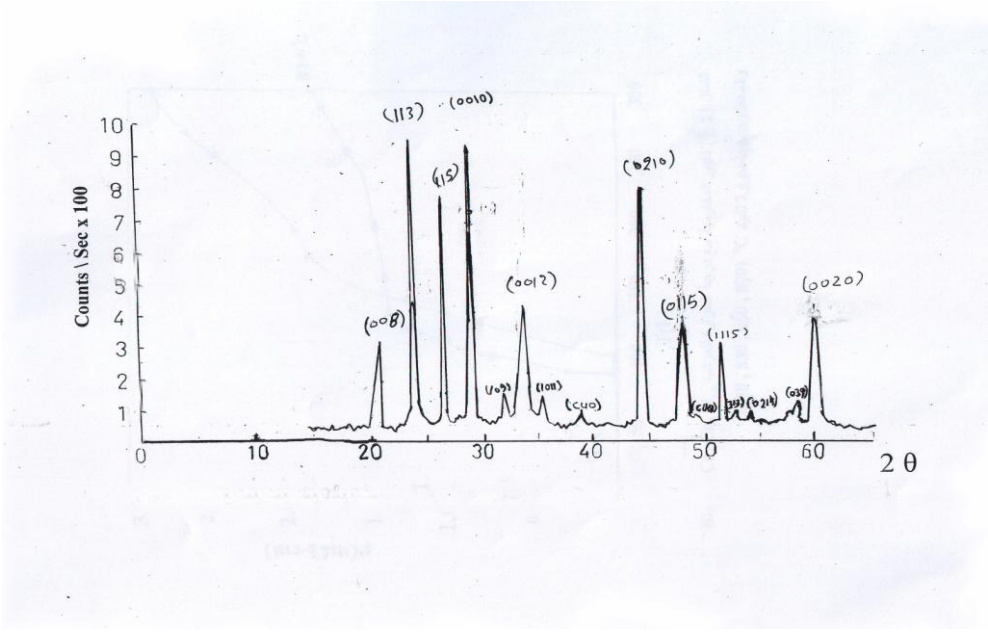

Fig(A-1):X-ray diffraction of($Bi_2Ba_2Ca_2Cu_3O_7$-δ)at(790)C°



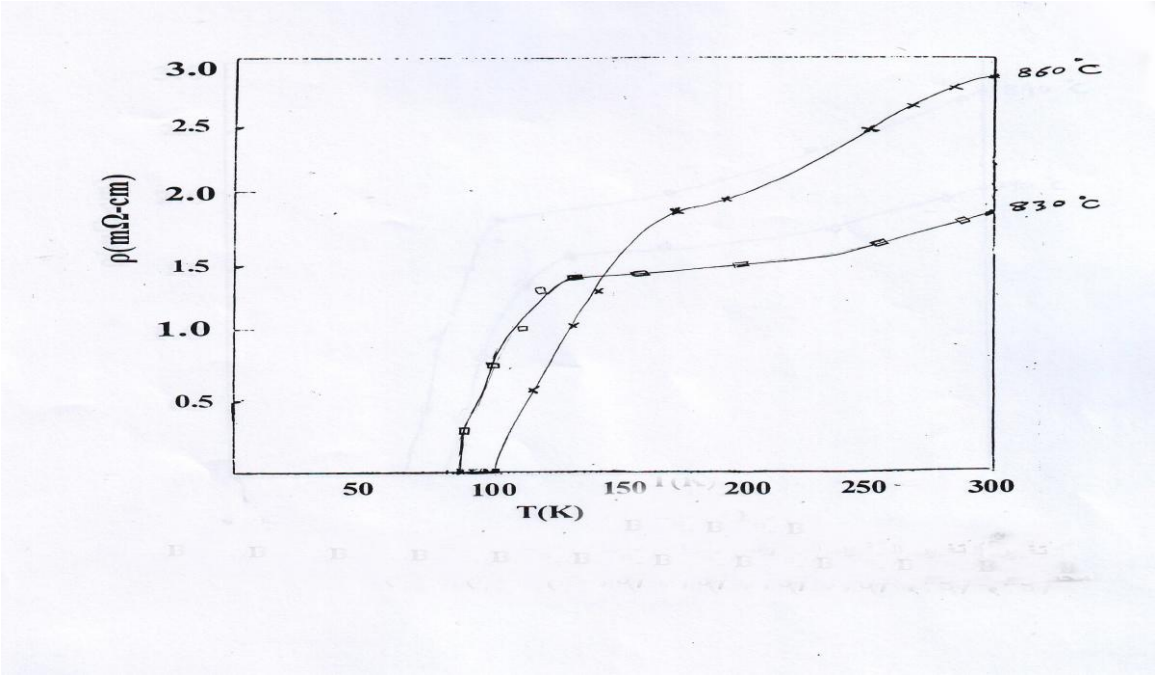

Fig(A-2):X-ray diffraction of ($Bi_2Ba_2Ca_2Cu_3O_7$-δ)at(830)C°



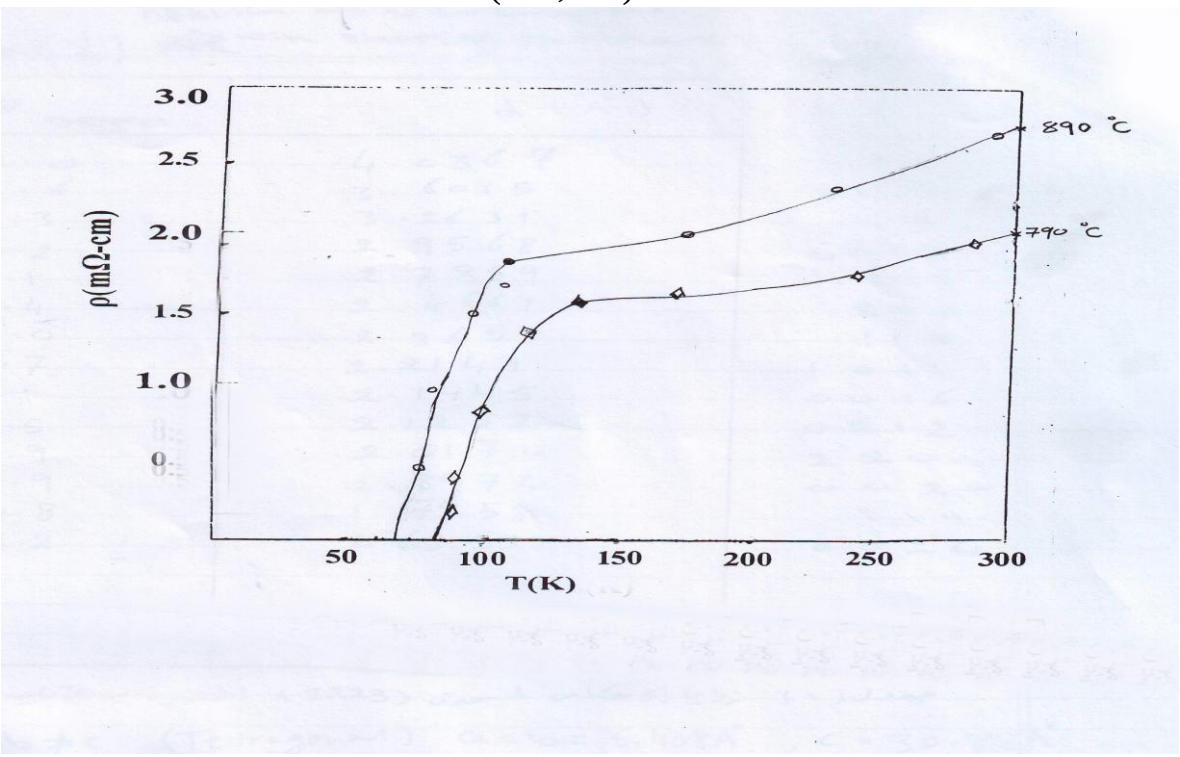

Fig(A-3)X-ray diffraction of ($Bi_2Ba_2Ca_2Cu_3O_7$-δ)at(860)C$^o$




Fig(A-4):X-ray diffraction of($Bi_2Ba_2Ca_2Cu_3O_7$-δ)at(890)C$^o$




Fig(B-1):Variation of resistively with temperature for($Bi_2Ba_2Ca_2Cu_3O_7$-δ) at(790,890)C°




Fig(B-2):Variation of resistively with temperature for($Bi_2Ba_2Ca_2Cu_3O_7$-δ) at(830,860)C°

Table(1):The atomic weight for compound(2223)

| The powder | The atomic weight ratio | The result (gm/1000) | The weight |
|---|---|---|---|
| $2(BiCO_3)$ | 2(209+12.01+16.0*3)=538.02 | 0.538 | W1 |
| $2(BaCO_3)$ | 2(137.34+12.01+16.0*3)=394.7 | 0.394 | W2 |
| $2(CaCO_3)$ | 2(40.1+12.01+16.0*3)=200.22 | 0.200 | W3 |
| 3(CuO) | 3(63.5+16.0)=238.6 | 0.238 | W4 |

Table(2):Reflection Angles for compound($Bi_2Ba_2Ca_2Cu_3O_{7-\delta}$)with sintering temperature at(860C˚)a=b≠c,(Tetragonal),a=b=5.408A˚,C=30.88A˚

| $2\Theta$ degree | D (A˚) | hkl |
|---|---|---|
| 22.0 | 4.0367 | 008 |
| 24.6 | 3.6015 | 107 |
| 27.3 | 3.2631 | 115 |
| 30.2 | 2.9568 | 0012 |
| 32.1 | 2.7859 | 109 |
| 36.4 | 2.4661 | 200 |
| 38.0 | 2.3659 | 110 |
| 40.7 | 2.2149 | 1011 |
| 42.1 | 2.1445 | 0016 |
| 42.5 | 2.1252 | 0212 |
| 44.9 | 2.0170 | 220 |
| 47.9 | 1.8974 | 0020 |
| 50.8 | 1.7958 | 119 |
| 56.2 | 1.6353 | 0024 |

Table(3):Reflection Angles for compound($Bi_2Ba_2Ca_2Cu_3O_{7-\delta}$)with sintering temperature at(890C˚)a≠b≠c,(orthorhombic),a=5.418A˚,b=5.429A˚, C=30.75A˚

| $2\Theta_{degree}$ | D (A˚) | hkl |
|---|---|---|
| 21.0 | 4.2867 | 008 |
| 23.5 | 3.7824 | 113 |
| 27.1 | 3.2875 | 115 |
| 28.4 | 3.1399 | 0010 |
| 31.2 | 2.8643 | 109 |
| 33.5 | 2.6727 | 0012 |
| 36.00 | 2.4926 | 1011 |
| 43.9 | 2.0562 | 0210 |
| 48.1 | 1.8900 | 0115 |
| 52.3 | 1.7477 | 1115 |
| 52.8 | 1.7323 | 313 |
| 53.9 | 1.5936 | 0214 |
| 59.2 | 1.5594 | 039 |
| 59.8 | 1.5452 | 00.20 |

Table(4):vaniation of (TC)with changing sintering temperature

| Sample | Sintering temperature(C) | Critical temperature.TC(K) |
|---|---|---|
| 2223 | 790 | 105 |
| 2223 | 830 | 115 |
| 2223 | 860 | 135 |
| 2223 | 890 | 92 |

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# تأثير درجة حرارة التلبيد على TC لمركب" $Bi_2Ba_2Ca_2Cu_3O_{7-\delta}$ "للنظام(2223)

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**الخلاصة**

حضر نماذج من المركب($Bi_2Ba_2Ca_2Cu_3O_{7-\delta}$)الفائق التوصيل عند درجات الحرارة العالية وذلك باستخدام طريقة تفاعل الحالة الصلبة تحت درجات حرارة التلبيد المختلفة هي.$C^{\circ}$ (790,830,860,890)واستخدمت تقنية حيود الأشعة السينية(X-ray) لدراسة التركيب البلوري للمركب وأظهرت النتائج إن المركب ($Bi_2Ba_2Ca_2Cu_3O_{7-\delta}$) ذات نظام (2223) والمحضر في درجة حرارة التلبيد .$C^{\circ}$ 860 ذات طور [Tetragonal] وبإبعاد الشبكية $A^{\circ}$ $a=b=5.408$ $C=30.77$ $A^{\circ}$ ولكن النماذج المحضرة عند درجة حرارة التلبيدة 890أظهرت الدراسة للمركب($Bi_2Ba_2Ca_2Cu_3O_{7-\delta}$) تحول في الطور من (Tetragonal )إلى( Orthorhombic ) بسبب تقلص المحور C وزيادة في المحورين (a,b) إذ إن قيم أبعاد الشبيكة

$a=5.418$ $A^{\circ}$ $b=5.429$ $A^{\circ}$ $C=30.75A^{\circ}$

واستخدمت طريقة قياس المقاومية الكهربا ئية لحساب قيم درجة الحرارة التحول (الحرجة) لهذه المركب من خلال استخدام منظومة التبريد التي تعمل بالنتروجين السائل وأظهرت المركب المحضر بدرجة حرارة التلبيد .$C^{\circ}$ 790 وذات درجة حرارة التحول
$TC=105K$ ولكن المركب المحضر في درجة .$C^{\circ}$ 830 أصبحت درجة حرارة التحول $TC=115K$

أما المركب المحضر بدرجة حرارة التلبيد .$C^{\circ}$ 860 فأن درجة حرارة التحول تساوي$K$ $TC=135$ وانخفضت الى $92K$ عند تحضير النموذج في درجة الحرارة .$C^{\circ}$ 890

وفسرت التغيرات في القيم (TC) درجة حرارة التحول (الحرجة) على أساس الانتظام في البنية البلوري الناتج عن زيادة في درجة حرارة التلبيد وكذلك زيادة نسبة الأوكسجين في المركب الذي تلعب دورا أساسيا في رفع درجة حرارة التحول (TC).