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Effect of repeated annealing on the synthetic and electrical properties of $Bi_{2-(x+y)}Pb_xAg_y\ Sr_2Ca_2Cu_3O_{10+\delta}$ electrically superconductor

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ABSTRACT

This research involved the preparation of $Bi_{2-(x+y)}Pb_xAg_y\ Sr_2Ca_2Cu_3O_{10+\delta}$ with different concentrations of (x,y) which are (x=y=0)(x=0.05,y=0.15)(x=y=0.1)(x=0.15,y=0.05) by the method of solid state reaction under hydrostatic pressure of 8 ton /cm^2 and at annealing temperature of 1123K in presence of sufficient amount of oxygen.
In order to obtain an increase in the regularity of crystalline composite and also to obtain the best critical temperature, the annealing was repeated on these samples at 723K.
After X-ray diffraction (XRD) test to know how effective the partial substitution and repeated annealing is, it was obvious that the crystalline composite was of tetragonal type and the best substitution ratio is when (x=0.05,y=0.15) where the values of lattice dimensions were a=b=5.4056°A, c=37.4226°A, after repeating the annealing process on these samples ,the crystalline composite kept its type and with conspicuous increase in the length of c-axis, but the best sample after repeating the annealing was at ratio of (x=y=0.1) where the values of lattice dimensions became a=b=5.4030°A,c=37.5230°A when it was a=b=5.4011°A,c=37.0660°A before repeating the annealing .When the electrical properties of these samples was studied ,we noticed that the oxygen has an important role in increasing the critical temperature and we obtained the best critical temperature T_c=144K and that's when x=0.05,y=0.15 while after repeating the annealing it was T_c=146K and that's when x=y=0.1 and it was T_c=136K before repeating the annealing.

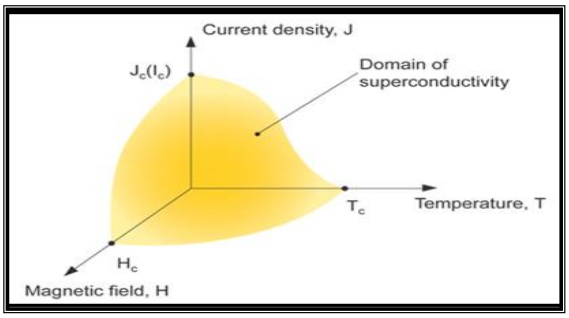
Introduction

The superconductive materials are those materials that allow the passage of electrical current without any resistance, and is characterized by the complete absence of magnetic field when it's cooled at a specific temperature which is called the critical temperature T_c. This property is important in many application of electronic science and medical tools [1,2]. It's possible to divide these materials into several divisions according to their T_c, nature of properties, and their structure: - classical superconductive materials like (Hg), organic superconductive material (K_3CO_6), heavy fermions ($CeCu_2Si_2$) superconductors that are not copper based ($BaPb_{-x}Bi_xO_3$), carbide compounds with superconductive property[3] ,and finally copper oxide based superconductors $Bi_2Sr_2Ca_2Cu_3O$ which is our subject and it's possible to add an amount of lead to these compounds[4] in order to have the highest regularity in the crystalline composite and also the best critical temperature Tc. There are many benefits and applications of superconductors such as; in electric energy transmission wires[5], magnetic resonance imaging (MRI) [6], super recognition radars[7], and flying trains[8].

2-Superconductors propertis

There are three factors that should come together in order to achieve the superconductive state which are:- critical temperature T_c, critical magnetic field H_c , and the critical current Jc , and each factor depends on the other two factors and in order to keep this superconductivity state these factors should stay in their critical values that can be illustrated on a critical surface as in figure (1) which shows that the highest value (J_c,H_c) happens at 0K ,while the highest value of T_c happens when the magnetic field H and the critical current J_c equals to zero[9].

These factors depend on the quality of the material, and it's the most important one in practical application ,and when there's an increase in any of the three values, the material loses its superconductive property.



Figure(1) critical surface

Materials and Methods

Samples of $Bi_{2-(x+y)}Pb_xAg_ySr_2Ca_2Cu_3O_{10+\delta}$ by a conventional solid state reaction method. The stoichiometric amounts of purity powders (99,9%) of Bi_2O_3 , $Sr(NO_3)_2$,CaO ,and CuO ,and Pb were added by weight (0.0)(0.15,0.05)(0.1,0.1)(0.05,0.15) The powders were mixed together by using a gate mortar for 15 minutes. The mixture homogenization takes place by adding a sufficient quantity of 2-propanol to form a paste during the process of grinding for about 30minutes. The mixture was grinded to a fine powder and then in air by using a tube furnace at 1123K for 24hr with rate of 393K/hr. The mixture then pressed into pellets of diameter 12mm and thickness of 1.2mm by using hydraulic press under pressure of 8 tons/cm^2.The pellets were annealed at 1123K for 72hr.then they're cooled down from 1123K to 873K at a rate of 293K/hr and it's left at this temperature for 12hr and then it's cooled from 873K to room temperature and at rate of 293K/hr .After having 4 samples of pellet like structures ,these samples were put in an electrical oven and its temperature was risen from room temperature to 723K and at rate of 393K/hr and they were left at this temperature for (2,4,6,8)hr in an atmosphere saturated with oxygen and then their temperature was cooled down from 723K to room temperature at a rate of 293K/hr to have the highest regularity in the crystalline composite.

Determintion of oxygen ratio (δ)

Conducting the titration process to determine the oxygen ratio (δ) in which an amount of 100mg is taken from the sample and is grinded very well using a small gate mortar to obtain a homogenous powder and with addition of isopropanol and then the powder is dried very well from the isopropanol alcohol .And it's placed in a conical flask after cleansing the flask from any other chemical material , and then 10ml of potassium iodide is added to the powder and 5ml of hydrochloric acid is also added to the powder .These solutions are mixed with the powder by placing a small magnetic bar in the flask and then placing it in a magnetic stirrer and then turning on the device in order for the magnetic bar to move inside the solution and helps in its mixing .After that, sodium thiosulfate solution with 0.015ml ratio is prepared. Upon addition of (HCL) and (KI) solution, the color of the solution changes to brown. Then, droplets of sodium thiosulfate is added until the color of the solution becomes dark brown. And at this state, the addition of droplets should cease. And then diluted starch is added and here the color of the solution will change from dark brown to dark blue. This indicates the liberation of iodine. After that, droplets of sodium thiosulfate are slowly added until the color of the solution changes and becomes colorless so the addition of the droplets ceases once the solution becomes colorless and so the titration process of the sample ends. Sodium thiosulfate that is added to the flask is recorded.

The oxygen ration is calculated as in the following equation.

$$\delta = \frac{[MA / MB] - (3\,ma / cv)}{[(2\,ma / cv)] - (M_0 / MB)}$$

Where (δ) represent the oxygen ration.

MA: Represent the molar mass of $Bi_{2-(x+y)}Pb_xAg_ySr_2Ca_2Cu_3O_{10+\delta}$.

MB: Represent the molar mass of sodium thiosulfate.

ma: Represent the weight of the sample.

Mb: Represent the sodium thiosulfate solution which we obtain from sodium thiosulfate concentration c=0.015gm/ml

mb=cv

v=Represent the sodium thiosulfate volume in the titration process.

C=concentration of sodium thiosulfate .

The volume of sodium thiosulfate that was used in titration process (mass = density× volume) V/ml× mg =c (gm/ml).

It was prepared by dissolving grains from the compound ($Na_2S_2O_3$) in distilled water with a ration of (0.015mg/ml), in this case the density (C) calculated and the finding the value of (δ) in the following equation [10]:-

$$\delta = \frac{[MA / MB] - (3\,ma / cv)}{[(2\,ma / cv)] - (M_0 / MB)}$$

Results and Discussion

a-study of synthetic properties of the compound.

the study of synthetic properties was done at 1123K and under hydrostatic pressure of 8 ton /cm^2 and the x-ray diffraction study revealed the regularity in the crystalline composite and using Bragg law $2d\sin\theta=n\lambda$ to calculate d_{hkl} which is the distance between parallel planes through reflection angles 2θ. Miller indices was found and using an application in the computer, values of dimensions of cell unit was found and it's tetragonal which means it has superconductive property[11,12] in which the values of dimensions were a=b=5.4340°A ,c=36.7887°A when x=y=0.

The results of x-ray diffraction revealed that during the substitution of x=0.05,y=0.15 the crystalline composite keeps its tetragonal type and appearance of conspicuous peaks and increase in length of c-axis

and values of lattice dimensions were a=b=5.4056°A, c=37.4228°A, but when x=y=0.1 is substituted we notice a decrease in length of c-axis but the crystalline composite keeps its type and this is a proof that a substitution at this ratio led to irregularity in crystalline composition and values of lattice dimension were a=b=5.4011°A,c=37.0660°A but at substitution of x=0.15 ,y=0.05 ,it turned out that the crystalline composite is regular and appearance of peak of narrow bases and a conspicuous increase in c-axis length as seen in figure (2) and values of lattice parameter were a=b=5.4037°A, c=37.4019°A as seen in table(1). After repeating the annealing on these samples, we noticed an improvement in the crystalline composite as seen in figure (3) and a conspicuous increase in the length of c-axis at a ratio of x=y=0 where the values of lattice dimensions became a=b=5.4219°A,c=37.0454°A. While at substitution ratio of x=0.05,y=0.15 and the increase is clear in the length of c-axis where a=b=5.4128°A,c=37.4815°A. And also at these two ratios (x=y=0.1),(x=0.15,y=0.05)where the values of lattice dimensions became (a=b=5.4030°A, c=37.5230°A) (a=b=5.4190°A,c=37.5022°A) as seen in table(2).

Table (1) Axes values a,b,c for the compound $Bi_{2-(x+y)}Pb_xAg_y\ Sr_2Ca_2Cu_3O_{10+\delta}$

Pb	Ag	ρ_m (g/cm^3)	a (Å)	c (Å)	c/a
0	0	1.6241	5.4340	36.7887	6.7700
0.05	0.15	1.5440	5.4056	37.4226	6.9230
0.1	0.1	1.6228	5.4011	37.0660	6.8627
0.15	0.05	1.6298	5.4037	37.4019	6.9215

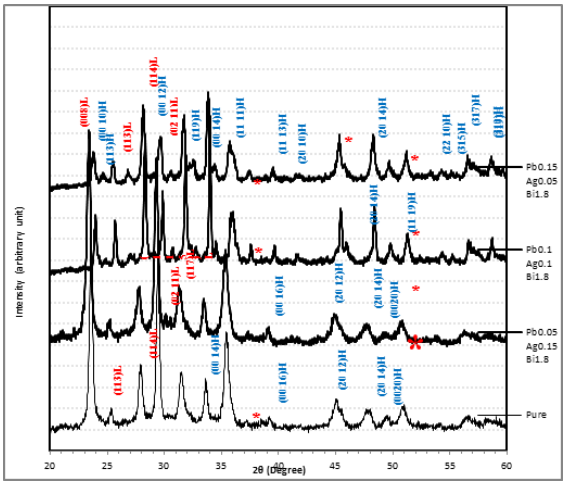


Figure (2)X-ray diffraction for compound $Bi_{2-(x+y)}Pb_xAg_y\ Sr_2Ca_2Cu_3O_{10+\delta}$

Table(2) Axes values a,b,c for the compound $Bi_{2-(x+y)}Pb_xAg_y\ Sr_2Ca_2Cu_3O_{10+\delta}$ After repeating the anneling

Pb	Ag	ρ_m (g/cm^3)	a (Å)	c (Å)	c/a
0	0	1.6392	5.4219	37.0454	6.8325
0.05	0.15	1.5504	5.4128	37.4815	6.9246
0.1	0.1	1.5472	5.4030	37.5230	6.9448
0.15	0.05	1.5601	5.4190	37.5022	6.9205

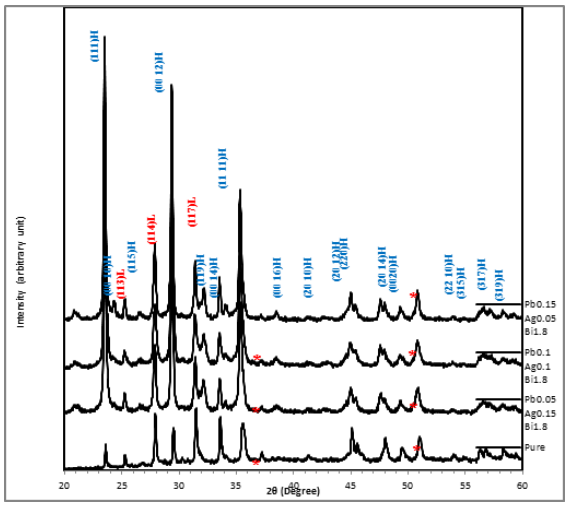


Figure (3)X-ray diffraction for compound $Bi_{2-(x+y)}Pb_xAg_y\ Sr_2Ca_2Cu_3O_{10+\delta}$ After repeating the anneling.

b- Study of electrical properties .

the study of electrical properties of the compound was done before and after partial substitution of Pb, Ag elements in Bi element and the T_c was 128K when x=y=0 and upon substitution by(x=0.05 y=0.15) the T_c increased to 143K and this can be explained by saying that the substitution at this ratio lead to increased grained volume and increase regularity in crystalline composite with an increase in oxygen lead where the compound was perfect at this substitution while substitution at a rate x=y=0.1 as seen in figure(4), we noticed a decrease in T_c where it was 136K and this explains irregularity in crystalline composite and the decrease in c-axis length and led to decrease in T_c and upon substitution at rate of (x=0.15, y=0.05) the T_c increase up to 140K as seen in table(3) .After repeating the annealing on these samples the critical temperature increased T_c=130K,144K,146K,142K according to the stated ratios as seen in figure(5) and in table (4) .

Table (3) relation between ratio substitution critical temperature and oxygen level after repeating the anneling.

sequence	Wt%	Tc K	δ
1	X=y=0	128	10.12
2	X=0.05,y=0.15	143	10.35
3	X=0.1,y=0.1	136	10.29
4	X=0.15,y=0.05	140	10.38

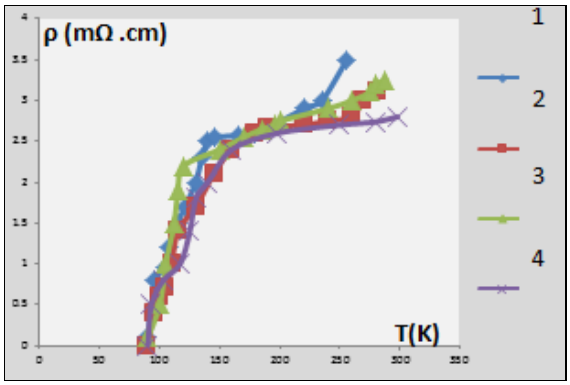


Figure (4) ***relation between resistance &critical temperature of compound*** **the $Bi_{2-(x+y)}Pb_xAg_y Sr_2Ca_2Cu_3O_{10+\delta}$**

Table (4) relation between ratio substitution critical temperature and oxygen level after repeating the anneling.

sequence	Wt%	Tc K	δ
1	X=y=0	130	10.21
2	X=0.05,y=0.15	144	10.40
3	X=0.1,y=0.1	146	10.42
3	X=0.15,y=0.05	142	10.39

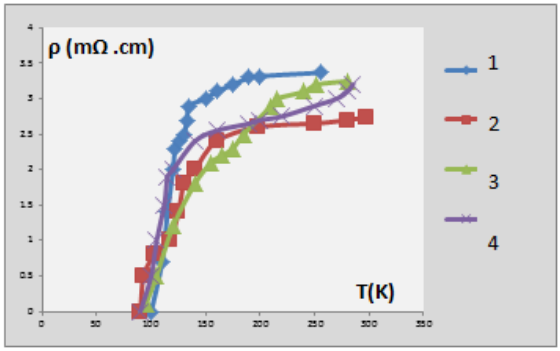


Figure (5): ***relation between resistance &critical temperature of compound*** **the $Bi_{2-(x+y)}Pb_xAg_y Sr_2Ca_2Cu_3O_{10+\delta}$ after repeating the anneling.**

Conclusion

The x-ray diffraction results revealed that the compound has a tetragonal crystalline composition and there is an increase in c-axis length with an increase of T_c, where the regularity in crystalline composite provides safe pathway for charge carriers cooper pairs in superconductors.

It is found that the partial substitution has an important role in increasing the critical temperature where T_c=128K,143K,136K,140K and that's when(x=y=0) (x=0.05,y=0.15) (x=y=0.1) (x=0.15,y=0.05). As it's known, the thermal treatment has an important role in the improvement of crystalline composite and after repeating the annealing, the critical temperature of the stated values became T_c=130K,144K,146K,142K.

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تأثير التلدين المتكرر على الخصائص التركيبية والكهربائية $Bi_{2-(x+y)}Pb_xAg_y\ Sr_2Ca_2Cu_3O_{10+\delta}$ للمركب الفائقة التوصيل الكهربائي

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الملخص

تضمنت هذه الدراسة تحضير العينات من المركب $Bi_{2-(x+y)}Pb_xAg_y\ Sr_2Ca_2Cu_3O_{10+\delta}$ وبتراكيز مختلفة ل x,y اذ كانت (x=y=0) (x=0.05,y=0.15) (x=y=0.1) (x=0.15,y=0.05) وبطريقة تفاعل الحالة صلبة وتحت ضغط هيدروستاتيكي 8 ton/cm^2 وعند درجة حرارة التلدين 1123K وبوجود كمية كافية من أوكسجين .

ومن أجل الحصول على زيادة الانتظام في التركيب البلوري ومن أجل الحصول على أفضل درجة حرارة الحرجة فقد تم تكرار التلدين على هذه العينات عند درجة حرارة 723K .

وبعد إجراء فحص حيود الاشعة السينية (XRD) لمعرفة مدى تأثير التعويض الجزئي والتلدين المتكرر على الخصائص التركيبية والكهربائية وتبين ان تركيب البلوري من نوع (tetragonl)، وان أفضل نسبة تعويض هي عندما (x=0.05,y=0.15) حيث كانت قيم أبعاد الشبيكة a=b=5.4056°A , c=37.4226 °A، وبعد تكرار التلدين على هذه العينات بقي التركيب البلوري محافظا على نوعه مع زيادة واضحة في طول محور c، أما أفضل عينة بعد تكرار التلدين فكانت عند النسبة (x=y=0.1) حيث أصبحت قيم أبعاد الشبيكة a=b=5.4030°A , c=37.5230°A حيث كانت قبل تكرار التلدين عند هذه النسبة a=b=5.4011°A , c=37.0660°A .

كما تمت دراسة الخصائص الكهربائية لهذه العينات وتبين أن للأوكسجين دور كبير في زيادة درجة الحرارة الحرجة وقد تم حصول على درجة حرارة T_c=144K وذلك عندما (x=0.05,y=0.15) .أما بعد تكرار التلدين فكانت T_c=146K وذلك عندما (x=y=0.1) حيث كانت قبل تكرار التلدين T_c=136K .