Effect of sintering time on $Bi_{1.6}Pb_{0.4}Sr_{1.8}Ba_{0.2}Ca_2Cu_3O_{10+\delta}$ compound quenched in air and liquid nitrogen

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Abstract:

High temperature superconductor with nominal composition $Bi_{1.6}Pb_{0.4}Sr_{1.8}Ba_{0.2}Ca_2 Cu_3O_{10+\delta}$ was prepared by solid state reaction method. Two sets of samples have been prepared .The first one was quenched in air; the second set was quenched in liquid nitrogen. X-ray diffraction analyses showed an orthorhombic structure with two phases, high –T_c phase (2223) and low-T_c phase (2212) in addition to that impure phase was found. It has been observed that quenched in air samples display a sharp superconducting transition and a higher-T_c phase than that of the quenched in liquid nitrogen samples.

Key words: cuperate superconductor, quenching, sintering time.

Introduction:

Since the discovery of BiSrCaCuO superconductor system, which has three different superconducting phases with similar crystal structure, great efforts were achieved to obtain high quality of Bi-2223 monophase. To get a nearly pure phase material, usually hundreds of hours were required (this involves repeated sintering steps). Such a long period of production is very disadvantageous for the practical application of this material. The partial substitution of Bi by Pb is well known to be the most efficient way of promoting the formation of this high –Tc superconducting phase [1]

Xu et al. [2] (1990) studied the superconductivity and microstructure of $Bi_{1.92}Pb_{0.48}Sb_{0.1}Sr_2Ca_2Cu_{3.2}O_x$ and $Bi_{1.8}Pb_{0.3}Sb_{0.1}Sr_2Ca_2Cu_3O_y$ samples, they found that different superconducting behavior due to different sintering conditions and were controlled by powder grain size and chemical activity.

Kocabas [3] (1998) prepared the samples by used the solid state reaction, The sample ($Bi_{1.5}Pb_{0.4}Sb_{0.1}$ $Sr_2Ca_2Cu_3O_y$) were sintered at 850°C for 60, 80 and 100 hours in air and then furnace cooled to room temperature. He was found the high Tc at 108, 110, and 112K, respectively. It is possible to say that the volume fraction of the high T_c phase increases with increasing sintering time and it appears to be the dominant phase in these ceramics.

Fernando et al. [4] (2006) studied the $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_y$ system prepared by solid state reaction techniques, the samples were prepared under different conditions. They found that conventional solid state reaction method requires very long heat treatment with several intermediate grinding stages in order to produce single Bi 2223 superconducting phase. Azam et al. [5] (2009) presented the effect of heat treatment and sintered time (80, 100 and 140)h on the properties of high-T_c Bi based superconductor. They observed that T_c (zero) obtained by this composition

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was (90 K , 100 K and 140 K) respectively.

The effect of substitution of Ni on Cu in $(Bi_{0.8}Pb_{0.2})_2(Sr_{0.9}Ba_{0.1})_2$ $Ca_2Cu_{3-x}Ni_x$ $O_{10+\delta}$ for (x=0, 0.1….1, 2,3) superconductor system and sintering time has been investigated by Thabit et al.[6] .The samples were prepared by solid-state reaction methods. The highest transition temperature (T_c) obtained was 113 for x=0.8.

To examine the effect of Ni substitution, mechanical properties (Vickers microhardness, Young's modulus, yield strength, fracture toughness and surface energy) by Vickers microhardness measurements have been carried out by Hermiz et al.[7] . The results showed a deterioration of the mechanical properties with the enhancement of Ni content .Mechanical properties of the samples are also found to be load dependent.

The aim of this work is to investigate the effect of preparation method and sintering time on the superconducting properties of $Bi_{1.6}Pb_{0.4}Sr_{1.8}Ba_{0.2}Ca_2$ $Cu_3O_{10+\delta}$ compound.

Material and Methods:

A conventional solid-state reaction was used to prepare the samples, appropriate amounts of Bi_2O_3 (99.9 %), Pb_3O_4 (99.9 %), $SrCO_3$ (99.9 %), $BaCO_3$ (99.9 %), CaO (99.9 %)and CuO (99.9 %) powders which were used as starting materials. The powders are with molar ratio of [Bi]:[Pb]:[Sr]:[Ba]:[Ca]:[Cu] = 1.6:0.4:1.8:0.2:2:3.

The powder of precursor was mixed together by using agate mortar. The mixture homogenization was achieved by adding a sufficient quantity of 2-propanol to form a past during the process of grinding for about (1 h). In the second step, the materials were calcined in air at 800 ◦C for (24) h and after that the mixture was pressed into pellets (1.3 cm) in diameter and (0.2) cm thick, using hydraulic type (SPECAC), under pressure of 0.5 GPa. The pellets were sintered in air at 850°C for 120,140,160 and 180h.

In this work two sets of samples were produced. The first set was quenched in air and the second set was quenched in liquid nitrogen.

Result and Discussion:

The x-ray diffraction patterns of samples quenched in air and liquid nitrogen sintering in different times are shown in Figs.(1-3).

Figs. indicate several peaks for high-T_c phase (Bi-2223) with amount of low-T_c phase (Bi-2212).with some impurity phases like Ca_2PbO_4 and $Sr_2Ca_2Cu_7O_\delta$ are detected at 2θ equal to 17.8,31.96 and 36.8 respectively[8,9].The present of Ca_2PbO_4 was known to result in a liquid phase which accelerated the anisotropic growth of high T_c phase [8]

It is clearly revealed that the heat treatment will improve the crystallites, the peaks to be sharper and an increase of peaks intensity. The most intense peak pattern of samples belongs to the high-T_c phase which also indicates increase in the volume fraction of the high-T_c phase with increasing sintering time [10].

Another feature was observed from the XRD patterns for the samples prepared by quenched in liquid nitrogen. The peaks have lower intensity in comparison with quenched in air and that the low-T_c phase (Bi-2212) was a dominate phase. This may attribute to the variation of oxygen content.

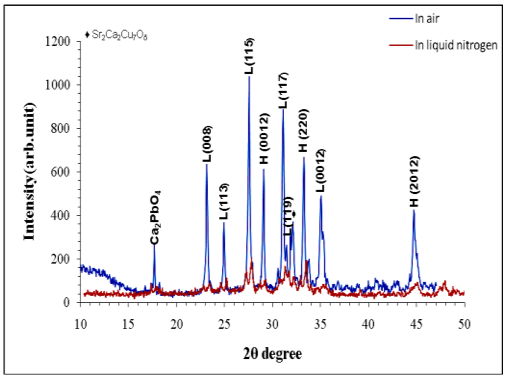

Fig.(1): X-ray diffraction patterns for $Bi_{1.6}Pb_{0.4}Sr_{1.8}Ba_{0.2}Ca_2Cu_3O_{10+\delta}$ samples sintered at 850 ^{o}C for 120 h quenched (a):air,(b): in liquid nitrogen.

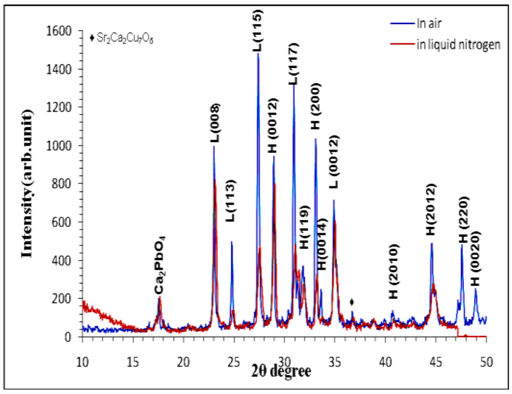

Fig.(2): X-ray diffraction patterns for $Bi_{1.6}Pb_{0.4}Sr_{1.8}Ba_{0.2}Ca_2Cu_3O_{10+\delta}$ samples sintered at 850 ^{o}C for 140 h quenched (a):air,(b): in liquid nitrogen.

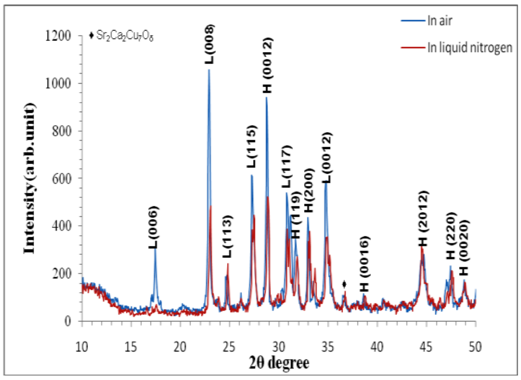

Fig.(3): X-ray diffraction patterns for $Bi_{1.6}Pb_{0.4}Sr_{1.8}Ba_{0.2}Ca_2Cu_3O_{10+\delta}$ samples sintered at 850 ^{o}C for 160 h quenched (a):air,(b): in liquid nitrogen.

The parameters *a,b,c* and *c/a* were also calculated from the XRD analysis as shown in Table (1) .This Table indicates that an increase of the sintering time from 140 h to 160 h change the structural parameters, the change in lattice parameters effect on the volume of the unit cell and then causes an increase of the density. The deformation in the c-axis adjusts the amount of charge transfer from Bi-O layer to Cu-O layer sheet will tend to improve the critical temperature. The difference in the cooling conditions is expected on the oxygen absorption behavior which will subsequently change the crystalline phases of superconductors [11].

The resistivity behavior as a function of temperature for quenching samples in air at different sintering time is shown in Fig.(4). It is observed from this figure. that prolonged sintering time up to a certain maximum time could improve the critical temperature (T_c) of stoichiomerty composition , it is clear that the transition temperature increases from 94 K to 100 K with the increase of the sintering time from 120h to 140h. When the sample sintered for 160h there is the decrease in a critical temperature to 89 K. The resistivity decreases slowly (it behaves like a superconductor) but does not become zero even at the boiling point of liquid nitrogen when the sintering time increases to 180 h. Similar behavior of the transition temperature with sintering time was found for samples sintered in liquid nitrogen with a transition temperature listed in Table (1) and Fig.(5) .This result indicates that the high T_c phase appears to be the dominant phase with the increasing of the sintering time up to 140h. This phase started to drop at more than this time. The increase in the T_c value is mainly due to the strong link and increasing of the contact areas between the grains during the sintering process time. Also Mawatari [12] suggested that the long sintering time will give a chance to insert extra layers of Cu-O_2 and Ca planes into the perovskite layers of the low-T_c phase. The reduction of the critical temperature can be due to either the crack formation as a result of excessive mechanical deformation, or to amorphous phase formation during the sintering process.

Indeed, samples quenched in air have sharp superconducting transition and higher T_c value than that quenched in liquid nitrogen. It has been suggested that the improved behavior of quenched samples is caused by an oxygen deficiency and this behavior could then result from the uptake of atmospheric oxygen.

Table (1): Variation in lattice parameters, c/a, density ρ_M and T_c values, for$(Bi_{0.8}Pb_{0.2})_2(Sr_{0.9}Ba_{0.1})_2Ca_2Cu_3O_{10+\delta}$ prepared by different methods.

Preparation Method	**Sintering time(h)**	**a Å**	**b Å**	**c Å**	**c/a**	**ρ_M(g/cm^3)**	**T_c K**
Sintering Time of quenching in air	120	5.417	5.419	37.140	6.856	1.573	94
	140	5.391	5.411	37.281	6.915	1.577	100
	160	5.413	5.412	37.058	6.846	1.580	89
	180	---	---	---	---	---	<77
Sintering Time of quenching in liquid nitrogen	120	5.432	5.46	37.178	6.844	1.556	91
	140	5.452	5.22	37.356	6.851	1.613	95
	160	5.461	5.83	37.194	6.810	1.448	<77
	180	5.37	5.43	37.137	6.915	1.584	<77

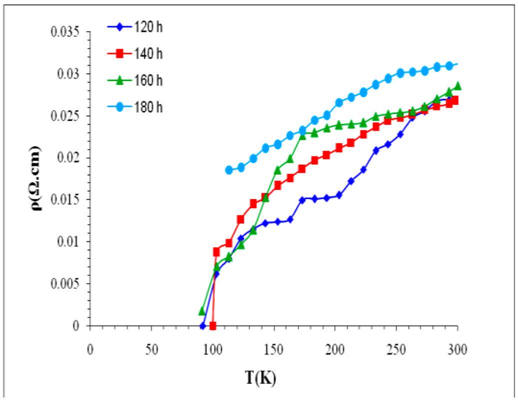

Fig.(4): Temperature dependence of resistivity for $Bi_{1.6}Pb_{0.4}Sr_{1.8}Ba_{0.2}Ca_2Cu_3O_{10+\delta}$ sintered at 850°C for various periods time quenched in air.

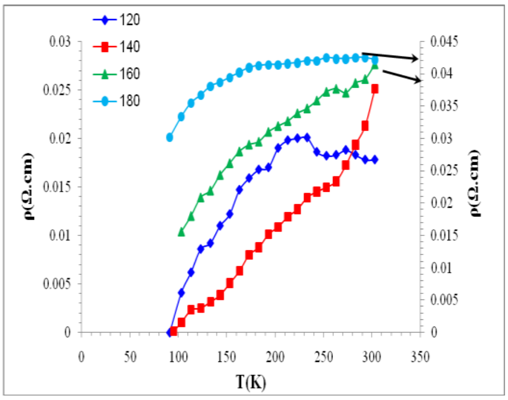

Fig.(5): Temperature dependence of resistivity for $Bi_{1.6}Pb_{0.4}Sr_{1.8}Ba_{0.2}Ca_2Cu_3O_{10+\delta}$ sintered at 850°C for various periods time quenched in liquid nitrogen

Conclusions:

1-It has been noticed that the preparation conditions play a great role in producing good superconducting samples. It is found that quenching the samples in air gave higher T_c value than that quenched in liquid nitrogen.

2- The optimum sintering time has been obtained for 140h, which gave us the best value of Tc for samples quenched in the two ways.

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تاثير زمن التلبيد على المركب $Bi_{1.6}Pb_{0.4}Sr_{1.8}Ba_{0.2}Ca_2Cu_3O_{10+\delta}$ المبرد فجائيا بالهواء والنتروجين السائل

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الخلاصة:

تم تحضير المركب $Bi_{1.6}Pb_{0.4}Sr_{1.8}Ba_{0.2}Ca_2Cu_3O_{10+\delta}$ الفائق التوصيل بطريقة تفاعل الحالة الصلبة.حضرت مجموعتين من العينات ودرست ظروف التبريد واختلاف زمن التلبيد حيث تم تبريد عينات المجموعة الاولى في الهواء بينما بردت عينات المجموعة الثانية في سائل النيتروجين

بينت تحليلات حيود الأشعة السينية ان جميع العينات لها تركيب المعيني القائم وبطورين، الطور الفائق التوصيل العالي (2333) والطور الفائق التوصيل الواطىء (2212) مع وجود طور الشوائب.

.ولوحظ ايضا ان العينات المبردة سريعاً بالهواء اظهرت انتقال حاد من حالة التوصيل الاعتيادي الى حالة التوصيل الفائق وبدرجة حرارة حرجة اعلى من العينات المبردة في سائل النيتروجين.