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The Effect of Sintering Duration Time and Cooling Condition on High T_c Phase of i-Sr-Ca-Cu-O Superconductor

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Abstract

High-T_c ceramic superconductor compound $Bi_2Sr_2Ca_2Cu_3O_{10}$ has been prepared by solid state reaction technique. Three sets of samples were prepared in order to study the effect of cooling conditions after sintering on superconductors, the first set of samples were (furnace cooled samples), the second set was (quenched in air), while the third set was (quenched in liquid nitrogen).

Four sintering duration time (50, 100, 150 and 200h) were used to determine the effective time that needed to increase the volume fraction of the high-T_c phase. It was found that the quenched in air samples show high- T_c and sharp superconducting transition, but the quenched in liquid nitrogen samples show low -T_c. The higher T_c obtained was 110 K and the proper sintering time was 150h. The x-ray diffraction pattern was used to identify the phases and the value of lattice parameters (a, b, and c).

الخلاصة

تم قياس التوصيلية الحرارية لمتراكبات من الايبوكسي المدعم باسود الكاربون والايبوكسي المدعم بالكرافيت لغرض دراسة تأثير اضافة اسود الكاربون والكرافيت على قيم التوصيلية الحرارية لهذه المتراكبات. عند زيادة نسبة الجسيمات المضافة فإن مقدار السلاسل الموصلة يزداد وهي بدورها تؤدي الى زيادة التوصيلية الحرارية لهذه المتراكبات.

Introduction

Soon after the discoveries of the oxide superconductor La-Sr-Cu-O in 1986 [1] with T_c of about 40K; Y-Ba-Cu-O [2] with T_c about 94K, as new superconductor was reported in 1987.

In 1988 cuprate superconductors become very well known. The discovery of superconductivity in Bi-System has generated great interest in the fields of condensed matter and material science. Maeda et al. [3] found T_c for the two superconductivity phases of Bi-Sr-Ca-Cu-O are $Bi_2Sr_2CaCu_2O_6$ which is called Bi-2212 and $Bi_2Sr_2Ca_2Cu_3O_{10}$ which is called Bi-2223 to be 80°K and 110° K respectively. The formation of the Bi-2223 phase is very narrow and often coexisting with Bi-2212 phase [4]. The desirable phase is the Bi-2223 phase because of its higher T_c. However, it is still difficult to synthesize its pure phase and the processing conditions required to form single Bi-2223 phase have yet to be determined [5, 6]. Consequently, the structure and the properties of this phase have not been studied extensively. The reason is that the formation of process taking place within a very limited temperature range.

Unless the difficulties in preparing Bi-2223 phase can be overcome phase. There is a little chance for scientific or technical progress in studies of this phase It is the aim of this work to synthesize an optimize 2223 phase within the system Bi-Sr-Ca-Cu-O and to find out the highest T_c that can be obtained out of this system.

Experimental Work

The samples with nominal composition $Bi_2Sr_2Ca_2Cu_3O_{10}$ were prepared by the solid state reaction method (SSR). The starting materials were of high-purity powders of Bi_2O_3 (99.9%), $Sr(NO_3)_2$(99.9%), $CaCO_3$ (99.9%) and CuO(99.9%) from BDH Chemicals Ltd., Poole England .

The elements in the system were in molar ratio Bi:Sr:Ca:CuO = 2:2:2:3 the constituents were mixed together manually by using a gate mortar, the mixture homogenization takes place by adding a sufficient quantity of high purity 2 – propanal during the process of grinding for 60 minute Then the mixture weighed and calcined in a tube furnace in the air for 10 hours at 800° C using a heating rate of 1°C/min , then cooled to room temperature with a cooling rate of 1°C/min., this procedure was used to remove the CO_2 and NO_2 from the mixture. Then grinding again the product powder of the mixture in a gate mortar for about 30 min. For further homogenous powder, then dried and pressed into pellets of 13 mm in diameter and (1.8-2)mm thick under a pressure of 5 ton/cm^2, using a manually hydraulic press. The pellets sintered in an air atmosphere at 870 °C with a heating rate 1°C/min.

The effect of sintering duration time on the critical temperature T_c values was investigated to determine the optimum sintering time required to obtain as high as possible T_c phase in the Bi-2223 compound. The prepared samples were sintered at temperature 870 °C and kept at this temperature for different sintering time 50h, 100h, 150h and 200h.

Three sets of samples were produced for each superconducting compound, the first set of samples were allowed to cool to room temperature in a furnace with a cooling rate 1 °C/min., these sample are called (furnace cooled samples). the second set of samples were removed from the furnace and rapidly quenched by immersion in liquid nitrogen, the second and third sets of samples are called (quenched samples).

The difference of the cooling conditions would indeed be reflected on the oxygen absorption behavior which will subsequently change the crystalline phases of superconductors.

Results and Discussion

The electrical resistivities of the furnace cooled samples are shown in Fig. (1). It is clear from this figure that the critical temperature T_c increases with the increase of the sintering time, when the sintering time 50h, T_c =83K, and when the sintering time 150h yielded higher- T_c superconducting phase of about 103K appeared more clearly with a long sintering time 150h, such result were also observed by Honda et al.[8], obviously, they suggested that longs sintering time is necessary to increase the high-T_c phase, and it can be said that long sintering time gives a chance to increase extra layers of Cu-O_2 and Ca planes into the layer structure of the low-T_c phase, because of the weak cohesive force with long c-axis of the high-T_c phase. However when the sintering time increases to 200h a decrease in the T_c values was obtained i.e. T_c=80K.

The values of the critical temperature T_c and the sintering duration time are summarized in Fig.(2).

The x-ray diffraction patterns of these samples are shown in Fig(3).We find that during the first sintering time (50h)Bi-2223 phase forms very slowly as indicated in the figure and the main peaks observed are corresponding to the Bi-2212 phase, with the prolongation of sintering time ((100-150)h), Bi-2223 phase increased sharply such that the peaks corresponding to the Bi-2223phase increased and became more sharp as the sintering time increases from 100h to 150h and more new peaks appeared i.e (0010, 119, 2014). For the sample sintered for 200h, the intensity of the peaks attributed to the low-T_c phase, became strong, this result indicates that the volume fraction of the low-T_c has increased when the sintering time increased above 150h.

The values of critical temperature and lattice parameter for these samples are listed in Table (1).

The temperature dependence of the resistivity for the quenched in air samples are shown in Fig.(4), from this figure we can see that the normal -state resistivity decreases with the increase of the sintering duration time, they showed sharp super conducting transition and higher- T_c value than other samples (furnace cooled and quenched in liquid nitrogen samples), on the other hand the highest T_c value obtained from these samples T_c =110K for the sample sintered at 870°Cfor 150h.

It has been suggested that the improved behavior of quenched samples is caused by an oxygen deficiency [9, 10], and this behavior could then result from the uptake of atmospheric oxygen, for more details of this work we refer to O'obodi [11].

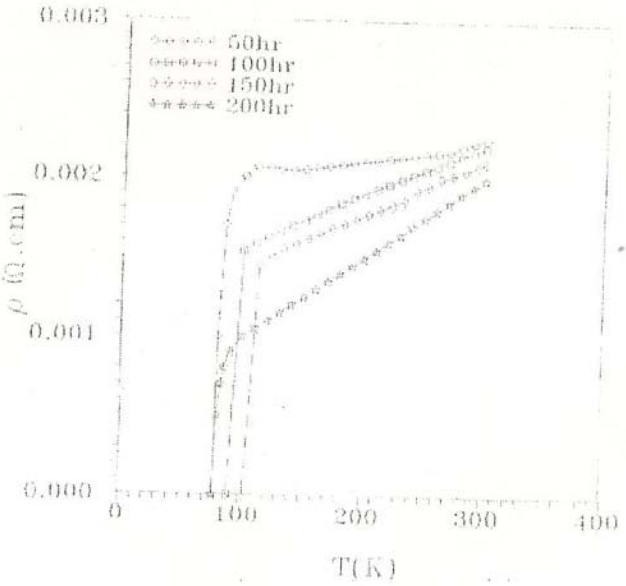

Fig.(1): Temperature dependents of the resistivity for the $Bi_2Sr_2Ca_2Cu_3O_{10}$ furnace cooled samples sintered at 870°C for various sintering times.

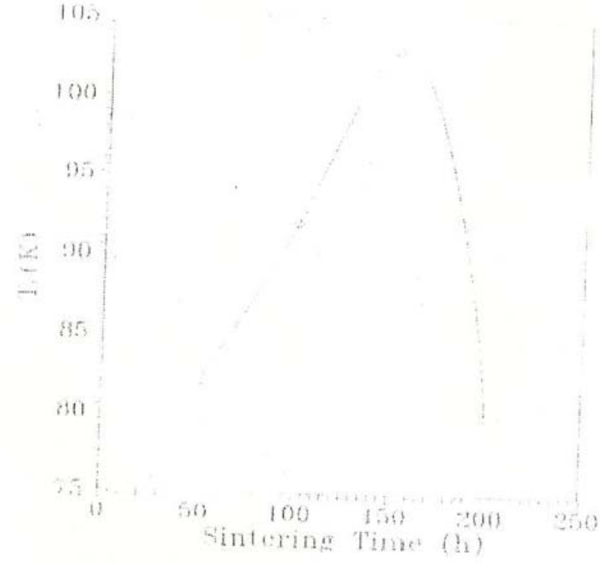

Fig.(2): Critical temperature of $Bi_2Sr_2Ca_2Cu_3O_{10}$ as a function of the sintering time.

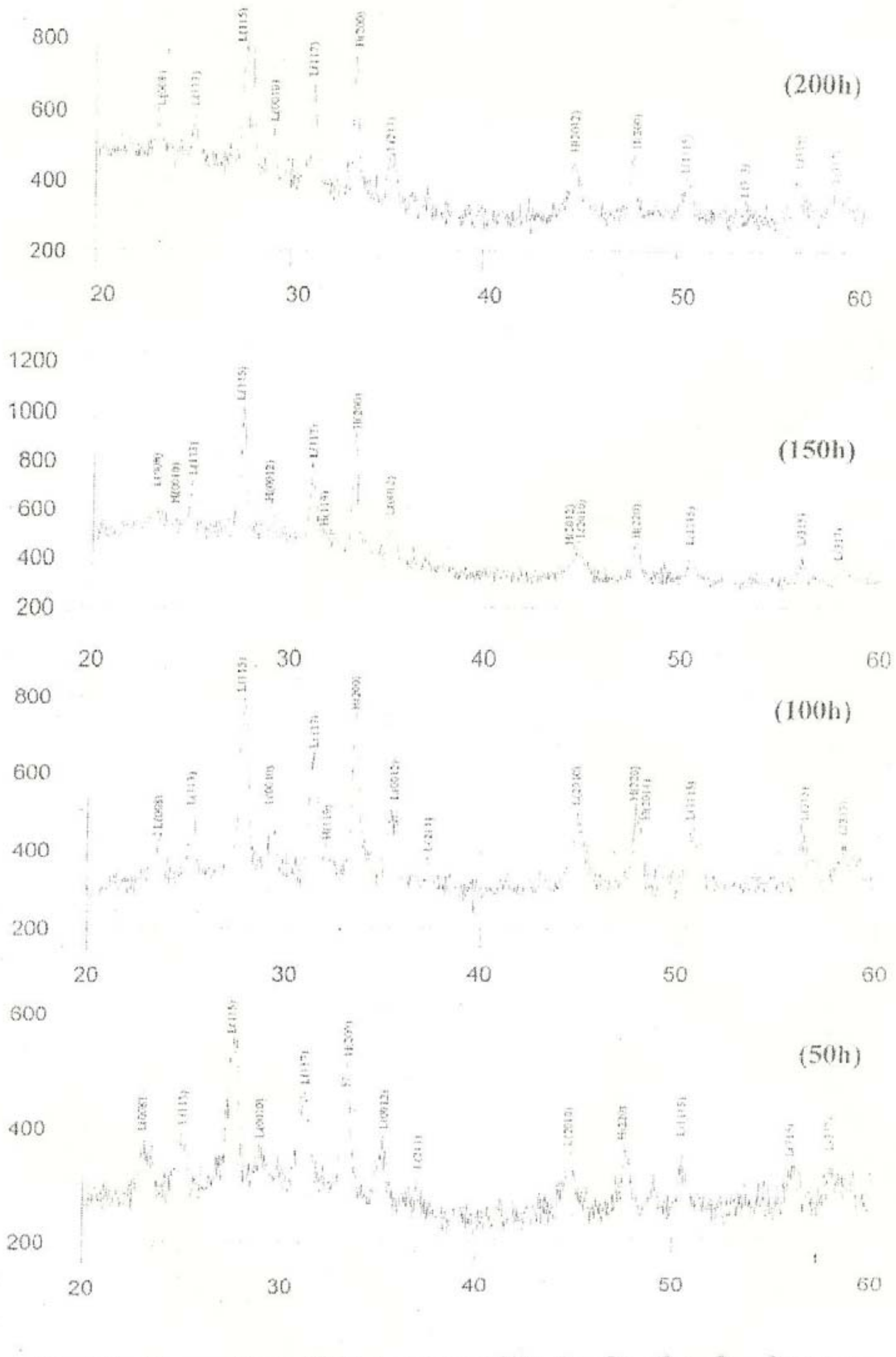

Fig.(3): X-ray diffraction patterns of $Bi_2Sr_2Ca_2Cu_3O_{10}$ furnace cooled samples sintered at 870°C for various sintering times.

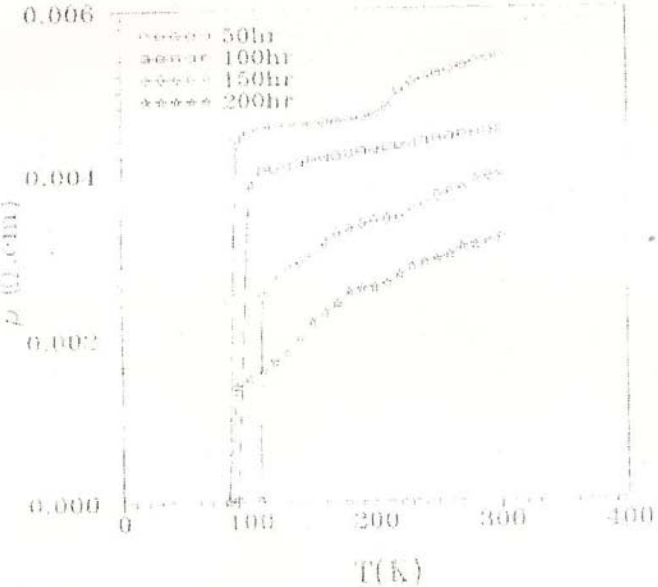

Fig.(4): Temperature dependents of the electric resistivities of quenched in air samples which were sintered at 870°C for various sintering times.

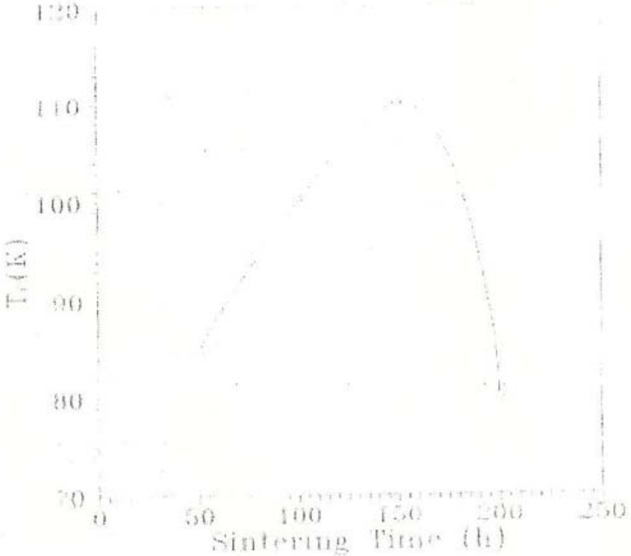

Fig.(5): Critical temperature of the quenched in air samples as a function of the sintering time.

Table (1): The lattice parameters and the critical temperature values at different sintering times.

Sintering temp.(°C)	Sintering time(hours)	a(A°)	b(A°)	c(A°)	T_c(K)
870	50	5.4172	5.4193	31.9612	82
870	100	5.4082	5.4082	34.1315	93
870	150	5.4130	5.4122	37.0672	103
870	200	5.4182	5.4201	31.8532	80

(Furnace cooled samples)

The x-ray diffraction patterns of these samples are shown in Fig. (6). Several peaks of Bi-2223 phase are observed in these samples, the quenching of samples in air enhanced their crystallinity, and it is noted that the main peaks of these samples are attributed to the 2223-phase with intensity higher than the other samples peaks.

The values of critical temperature and the lattice parameter, a, b and c for the quenched in air samples are listed in Table (2).

Comparing the lattice parameters of Tables (1)and (2) we observed the c-parameter of the samples quenched in air (Table(2)) to slightly longer than that furnace cooled samples (Table(1)), but much longer for the samples sintered for 100h and 200h. We can consider that CuO concentration planes has slightly increased, the criteria for that the general increase in T_c as the sintering time increases. The results in Table (1) and (2) of the lattice parameters and their variation are in agreement with the data of One et al[12].

Fig. (7) shows the results of electrical resistivity measurement for the quenched in liquid nitrogen samples. The resistance drop for all these samples occurred in two steps implying that samples are mainly of the two phases Bi-2212 and Bi-2223, except the samples sintered for 150h which shows super conducting transition as nearly high T_c phase, and their resistance drop occurred in one step. It can be said that prolonged sintering time to 150h was very effective in increasing the volume fraction of the high- T_c phase. The highest T_c in this set of samples were obtained at 93K for the samples sintered for 150h, while other samples exhibited super conducting transition around 80K. However, the transition at 110K was not observed for all samples of this set.

Table (2): The lattice parameters and the critical temperature values at different sintering times.

Sintering temp.(°C)	Sintering time(hours)	a(A°)	b(A°)	c(A°)	T_c(K)
870	50	5.4212	5.4220	31.9925	85
870	100	5.4078	5.4089	37.0451	100
870	150	5.4052	5.4052	37.0977	110
870	200	5.4206	5.4202	36.6541	80

(Quenched in air samples)

Table (3): The lattice parameters and the critical temperature values at different sintering times.

Sintering temp.(°C)	Sintering time(hours)	a(A°)	b(A°)	c(A°)	T_c(K)
870	50	5.4084	5.4108	30.8796	80
870	100	5.4211	5.4198	31.4652	85
870	150	5.4012	5.4012	34.4193	93
870	200	5.4296	5.4292	30.9082	80

(Quenched in liquid nitrogen samples)

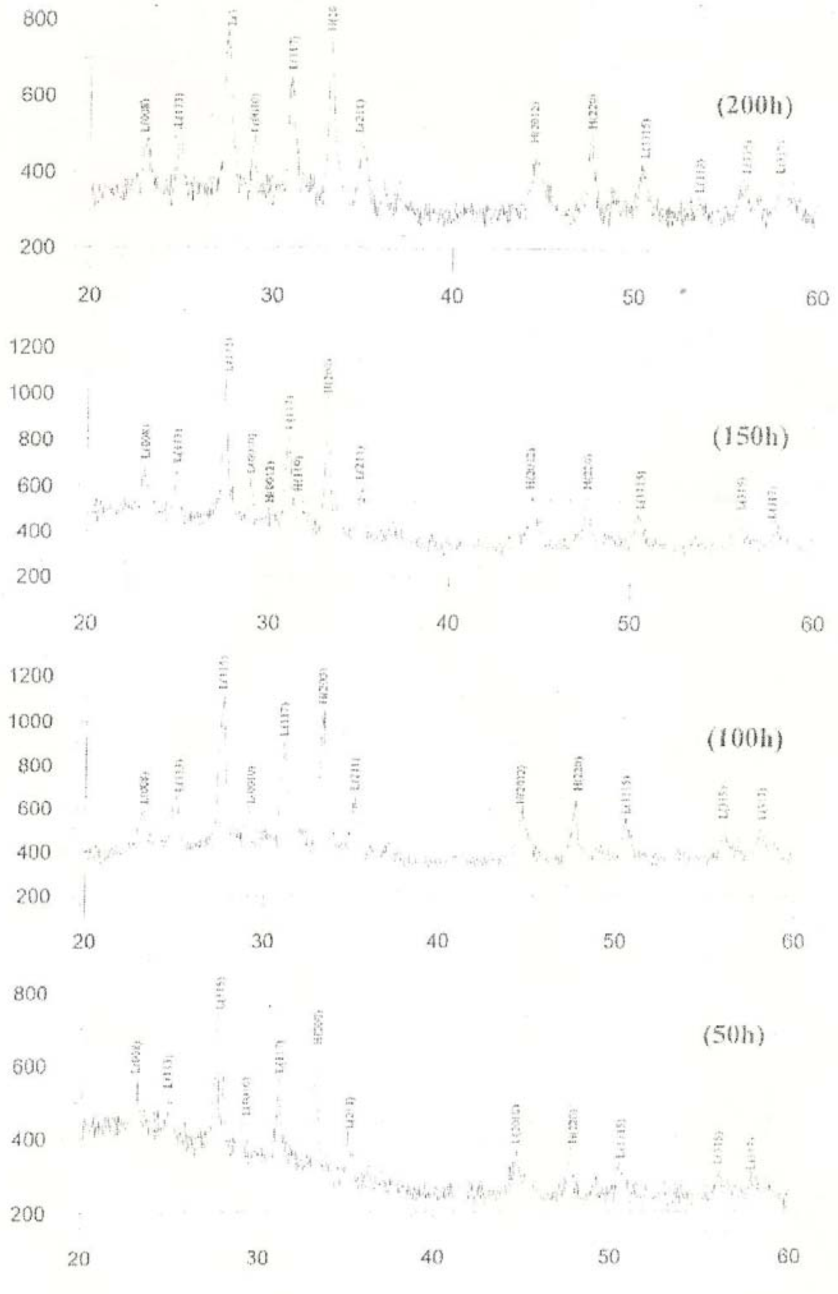

Fig.(6): X-ray diffraction patterns of $Bi_2Sr_2Ca_2Cu_3O_{10}$ quenched in air samples, sintered at 870°C for various sintering times.

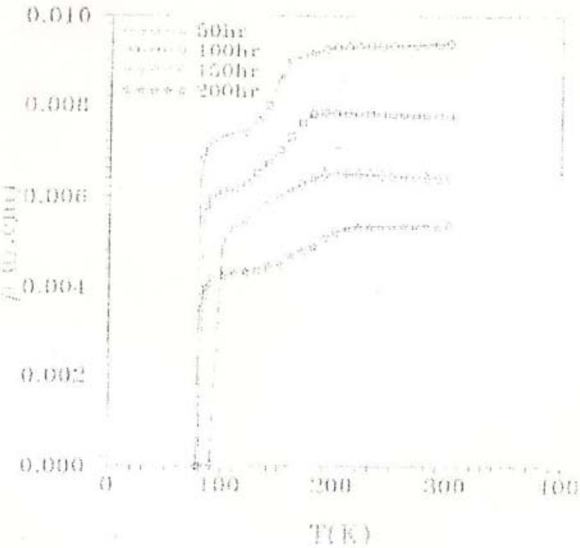

Fig.(7): Temperature dependents of the electric resistivities of the quenched in liquid nitrogen samples which were sintered at 870°C for various sintering times.

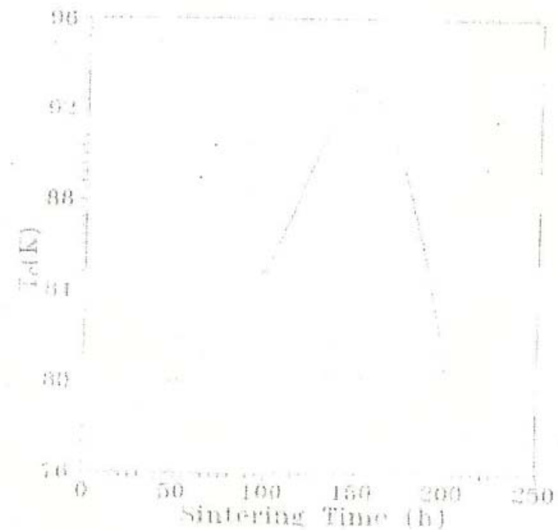

Fig.(8): Critical temperature of the quenched in liquid nitrogen samples as a function of the sintering time.

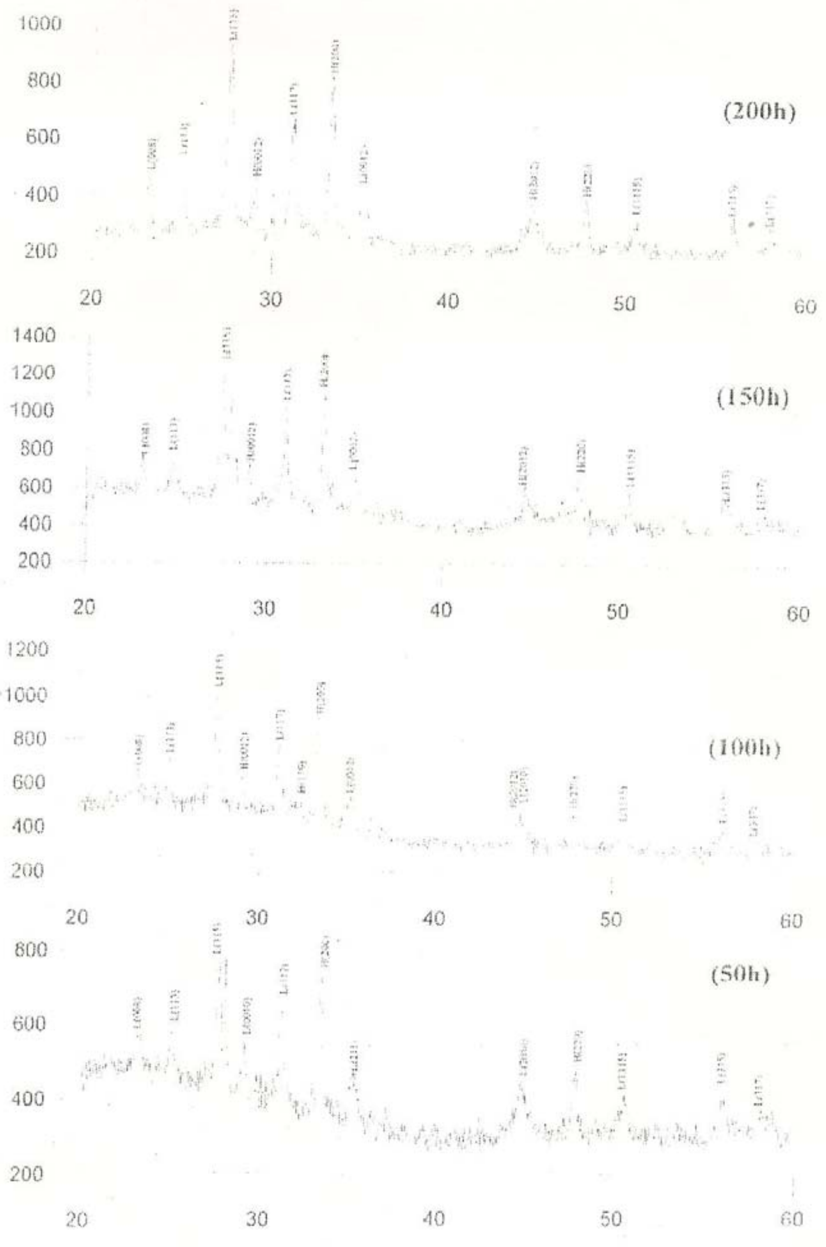

Fig.(9): X-ray diffraction patterns of $Bi_2Sr_2Ca_2Cu_3O_{10}$ quenched in liquid nitrogen samples, sintered at 870°C for various sintering times.

The critical temperature (T_c) is plotted versus the sintering duration time for these samples as shown in Fig. (8).

The x-ray diffraction patterns of these samples are shown in Fig. (9). It is seen from the figure that samples quenched in liquid nitrogen showed peaks with intensity higher than their counterpart shown in Figs.(3 & 6), see for example the prominent peaks (115), (117) of the low phase. However, several peaks of 2212 phase are observed. These results are found to be in agreement with the resistivity measurement results.

The values of critical temperature and the lattice parameter a, b and c for these samples are listed in Table (3).

Conclusion

From the results of this study we found the following conditions to obtain the optimum high-T_c phase:

The sintering time to obtain high-T_c phase 150h.

The x-ray analysis and resistivity measurement results indicated that the volume fraction of the high-T_c phase increased with the increase of sintering time up to a certain maximum time 150h and at sintering temperature 870°C, above these time and temperature the high-T_c phase started to drop and disappear to the account of lower T_c phases.

The formation of Bi-2223 phase by quenching the samples in air gave T_c 110K, which is higher than the T_c's of the quenched in liquid nitrogen samples and furnace cooled samples.

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