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EFFECT OF SINTERING TIME ON THE SUPERCONDUCTING PROPERTIES OF $Tl_{1.6}Hg_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$ and $Tl_{1.8}Bi_{0.2}Sr_2Ca_2Cu_3O_{10+\delta}$ COMPOUNDS

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ABSTRACT
The solid state reaction technique has been used to prepare homogeneous superconducting samples for $Tl_{1.6}Hg_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$ and $Tl_{1.8}Bi_{0.2}Sr_2Ca_2Cu_3O_{10+\delta}$. The effect of sintering time on the transition temperature (T_c) has been studied. The structural properties and electrical resistivity of these systems have been investigated using x-ray diffraction (XRD) and four probe method respectively. The XRD pattern showed a tetragonal structure, with at least two superconducting phases. It is found for Tl-2223 system, the T_c decreased with increasing sintering time.

KEYWORD:-High Temperature Superconductors, TBCCO System, Superconductor Properties**.**

INTRODUCTION
Superconductivity was first observed in mercury by the Dutch physicist Heike Kamerlingh Onnes in 1911[1]. For a long time, it has been known from conductivity experiments that the electrical resistance of a metal decreases with decreasing of temperature. Onnes was measuring the variation of the electrical resistance of mercury with temperature; he was amazing to find that at 4.2K the resistance suddenly dropped to zero. He called this effect superconductivity and the temperature at which it occurs is known as the critical transition temperature (T_c). In 1987, a research group led by Chu[2] discovered a superconductor compound at above the liquid nitrogen temperature. They used yttrium substitute for lanthanum in a 1:2:3 ratio with barium and copper ($YBa_2Cu_3O_{7-\delta}$). Bismuth and thallium based superconductors were discovered in 1988 [9] which became superconducting at 110K and 125K, respectively.

Dou et al (1988)[3] studied superconducting properties of Tl-Ba-Ca-Cu-O ceramics prepared by a solid state reaction under optimum conditions (880°C sintering in flowing O_2 for 3h). The resistivity, AC susceptibility, and Meissner effect were measured. The temperature-dependent resistivity of $Tl_2Ba_2Ca_2Cu_3O_{10+y}$ showed a degradation in T_c after increasing the sintering time from 3h to 6h and the temperature dependent AC susceptibility showed that at zero applied field the superconducting transition is reasonably sharp with T_c=110K.

Liu et al (1992)[4] studied the samples of $TlSr_2Ca_2Cu_3O_9$ system prepared by a solid state reaction, with replacing part of the Tl by Pb to get pure phase of $(Tl_{0.5}Pb_{0.5})Sr_2Ca_2Cu_3O_9$ which enhanced the high temperature superconductivity with $T_{c(onset)}$=130K, $T_{c(midpoint)}$=124K, $T_{c(zero)}$=122.5K as measured by electrical resistance, and the diamagnetic onset temperature $T_{c(mag)}$= 124K as determined by DC magnetic susceptibility. Jia *et al* (1994)[5,6] reported on the effect of substitution of Hg at the Tl sites in the oxygen deficient TlO_δ layer of $Tl_2Ba_2Ca_2Cu_3O_{10+\delta}$ cuprate superconductor. They prepared the samples by the two-step reaction process and they found that partial substitution of $Tl^{3+}$with Hg^{2+}in $Tl_2Ba_2Ca_2Cu_3O_{10}$ produces a stable Tl-2223 phase with the highest possible T_c and claimed that at room temperature the Hg-doped samples have a higher resistivity than the undoped specimen.

Bulk polycrystalline $Tl_2Ba_2CaCu_2O_x$ system has been fabricated by Ossandon *et al*. (2001)[7], results showed that the crystalline unit cell is tetragonal with lattice constant a=3.8550Å and c=29.318 Å (cell volume of $2.18\times10^{-23}m^3$) containing two sets of adjacent oxygen copper layers. The samples were irradiated at room temperature in air with 0.8 GeV protons. The superconductive properties of the virgin and irradiated materials were investigated magnetically.

Single crystals of TBCCO (Tl-2212) have been grown from a stoichiometric mixture of Tl_2O_3 and a precursor $Ba_2CaCu_2O_x$ prepared by Chowdhary *et al*. (2002)[8]. They observed that the transition temperature T_c=105K and the transition width ΔT_c was around 5K, as determined from the temperature-dependent magnetization (MT) measurements, at a field of 10G. Khan et al[9] have studied the enhanced Inter-grain Connectivity in $(Cu_{0.5}Tl_{0.5})Ba_2Ca_2Cu_3O_{10-\delta}$ Superconductors. Kareem and Tariq [10] have investigated the effect of simultaneous substitution of strontium at the barium site of $Tl0.6Pb_{0.4}Ba_{2-x}Sr_x Ca_2Cu_3 O_{9-\delta}$ Superconductors and found that $T_{c\ (off)}$= 113K for $Tl_{0.6}Pb_{0.4}Ba_{1.5}Sr_{0.5}Ca_2Cu_3O_{9-\delta}$.

EXPERIMENT
Two different types of superconducting systems according to their nominal composition $Tl_{2-x}Hg_xSr_2Ca_2Cu_3O_{10+\delta}$ and $Tl_{2-x}Bi_xSr_2Ca_2Cu_3O_{10+\delta}$ were prepared by a two step solid state reaction method .precursor $Sr_2Ca_2Cu_3O_{10+\delta}$ was first prepared using high purity powders of $Sr(NO_3)_2$, CaO and CuO as starting materials. Then, Tl_2O_3 and Bi_2O_3 were

added to the mixture and grinding them in agate mortar for about 30min to obtain a very fine and optimum homogenous powder. The mixtures were pressed into a pellet of (0.2-0.3)cm in thickness and 1.3cm in diameter, under a pressure of about 3 ton/cm^2 . The samples were sintered in air atmosphere of 860°C for 3h and 6h. The resistivity measurements were carried out by the four probe method with 30mA current .The structure of the prepared samples was obtained by using X-ray diffractometer (XRD) type Philips having the following features (source : $Cu_{K\alpha}$,voltage : 40kv , current : 20 mA, wavelength : 1.5405 Å). X-ray fluorescence type (OXFORD), having the following features (source: $Cu_{K\alpha}$, current: 10μA voltage: 26kV, time constant: 19.0s) was used to test the concentration of elements for some prepared samples.

RESULTS & DISCUSSION

In order to clarify the effect of prolonged sintering times on the electrical resistivity and the transition temperature (T_c), the prepared samples of nominal compositions $Tl_{2-x}Hg_xSr_2Ca_2Cu_3O_{10+\delta}$ and $Tl_{2-x}Bi_xSr_2Ca_2Cu_3O_{10+\delta}$ systems with x ranging from 0 to 1 have been sintered at 3h and 6h (Figs.1-4) . Initially the specimens sintered at 860^{o}C for 3h have a low resistivity and T_c of about 120K and 100K for $Tl_{1.6}Hg_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$ and $Tl_{1.8}Bi_{0.2}Sr_2Ca_2Cu_3O_{10+\delta}$ respectively. When the specimens are heat treated for an additional 3h, the superconducting properties degrade. Similar results have been reported by Dou *et al.* [11]for $Tl_2Ba_2Ca_2Cu_3O_{10+\delta}$ system sintered at 880°C with flowO_2. The ρ-T data indicate that the higher T_c phase (Tl-2223) dissociates giving rise to a second phase of composition (Tl-1223) with T_c=100K for $Tl_{1.6}Hg_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$ and 80K for $Tl_{1.8}Bi_{0.2}Sr_2Ca_2Cu_3O_{10+\delta}$. However T_c was observed to decrease after a longer thermal treatment. It has been claimed that such a decrease can be attributed to crack formation a result of excessive mechanical deformation[11]. In our view higher time could not lead to crack process but it comes nearer to a melting state as we mentioned before It is more probably the low temperature phase grows and is preferable at a state near the melting point as it is almost always the case [11]. We should point out also if any deformation acceptable would have taken place, it would rather enhance the polarization which helps for a better T_c. On the other hand drastic deformation leads to negative results, a thing which we believe would not happen.

The significant point which can be deduced from our data to explain why T_c decreased for longer period is that data of δ given in Table (1). It has been shown from other researchers and our work that the decreasing δ blow a certain limit, T_c degrades. Dou *et al.*[11] found that T_c for $Tl_2Ba_2Ca_2Cu_3O_{10+\delta}$ decreases as the sintering time increases. They attributed the decrease of T_c to the loss of thallium during the prolonged sintering treatment**.**

It is well accepted that the phonon coupling interaction (Cooper pairs) is necessary for T_c to take place. In case of degradation ,we can think of such reaction becomes weaker and resonant tunneling through localized centers along the c-axes between the CuO_2 planes across TlO plane or its dopant partner decreases .The reason is that for tunneling to take place some conditions should be satisfied first the transport across the 2D in the CuO_2 plane should be high enough to produce high electron density at Fermi level second small energy gap should be formed third Cu-O bond stretching phonon- coupling fluctuation should be low and the transport in the 2D should not be suppressed, fourth the Cu-O bonds must be ordered.

Moreover, x-ray diffraction patterns of the sample $Tl_{1.6}Hg_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$ are shown in Fig. (5), we find that during the first sintering time (3h) the main peaks observed are corresponding to the Tl-2223 phase, with the prolongation of sintering time (6h); the intensity of the peaks attributable to the low-T_c phase becomes stronger. This result indicates that the low-T_c phase have increased in these samples. The values of critical temperature and lattice parameter for these samples are listed in Table (1).

The XRF analysis (Figs.6) shows that, in sample b, the Tl content is relatively lower than in sample a. This may mean that Tl may be partially evaporated. However we should point out that the evaporation of O is most probable as our empirical data showed during the resintering process.

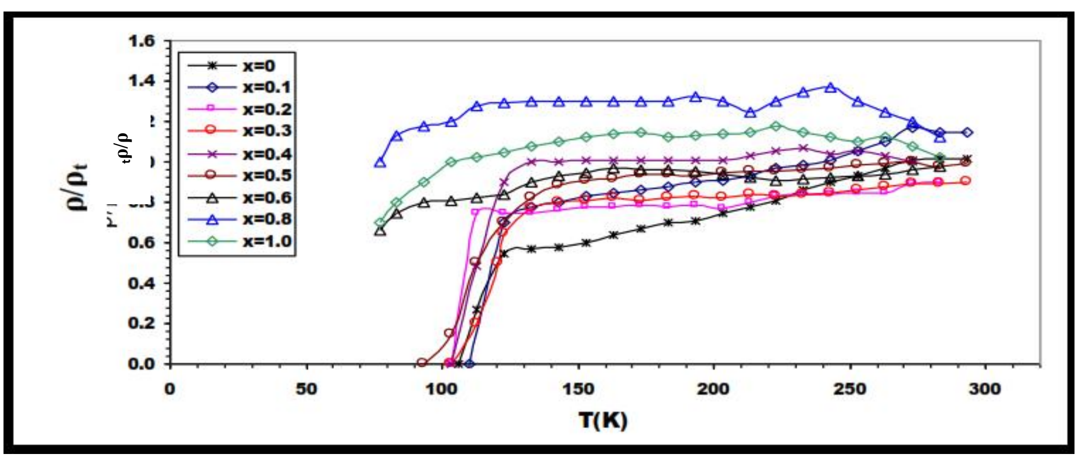

FIGURE 1: Temperature dependence of normalized resistivity for $Tl_{2-x}Hg_xSr_2Ca_2Cu_3O_{10+\delta}$ system sintered at 860°C for 3hours

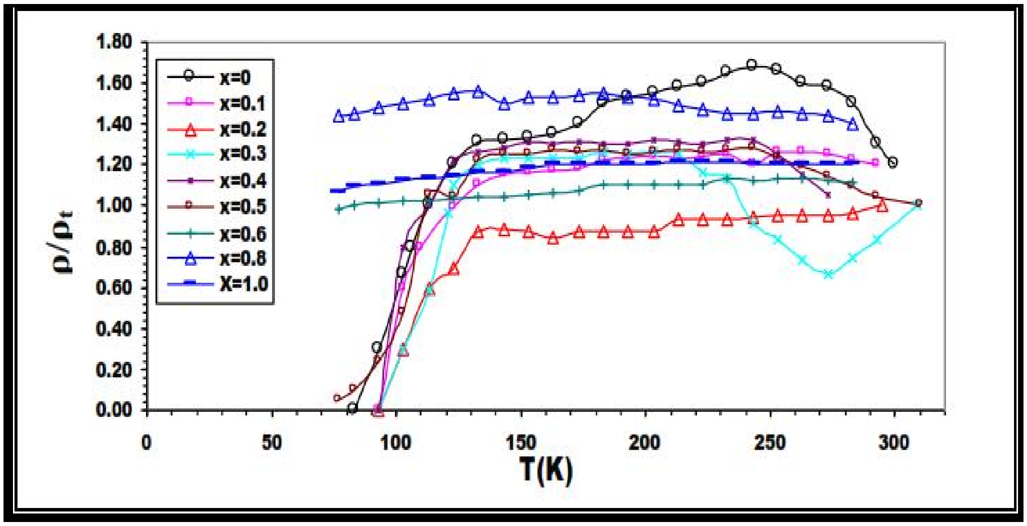

FIGURE 2: Temperature dependence of normalized resistivity for $Tl_{2-x}Hg_xSr_2Ca_2Cu_3O_{10+\delta}$ system sintered at 860°C for 6hours

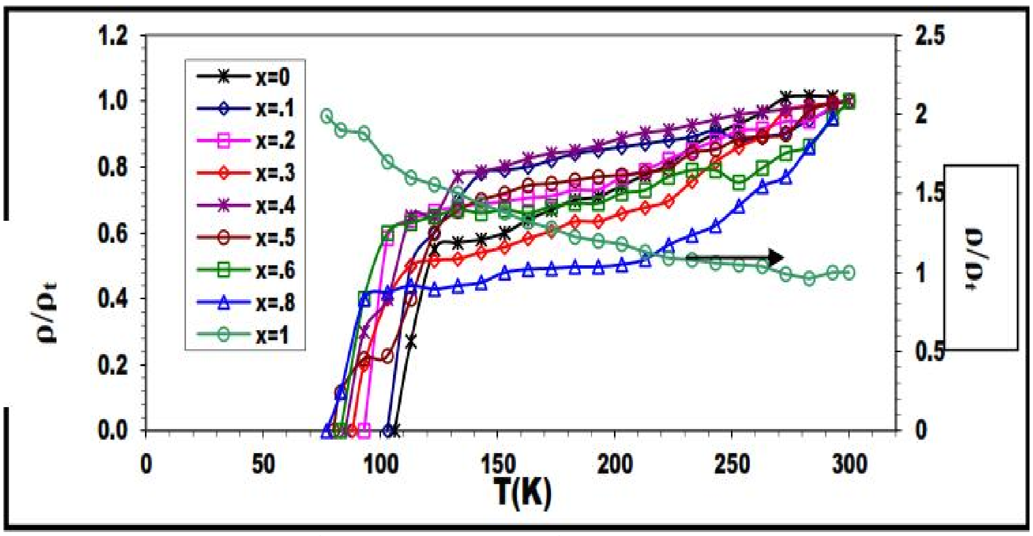

FIGURE 3: Temperature dependence of normalized resistivity for $Tl_{2-x}Bi_xSr_2Ca_2Cu_3O_{10+\delta}$ system sintered at 860°C for 3hours

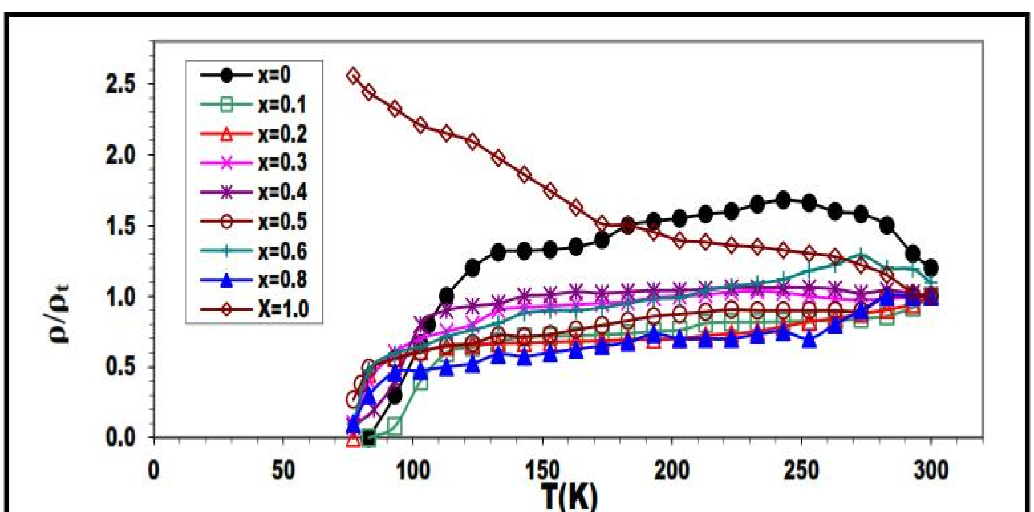

FIGURE 4: Temperature dependence of normalized resistivity for $Tl_{2-x}Bi_xSr_2Ca_2Cu_3O_{10+\delta}$ system sintered at 860°C for 6hours

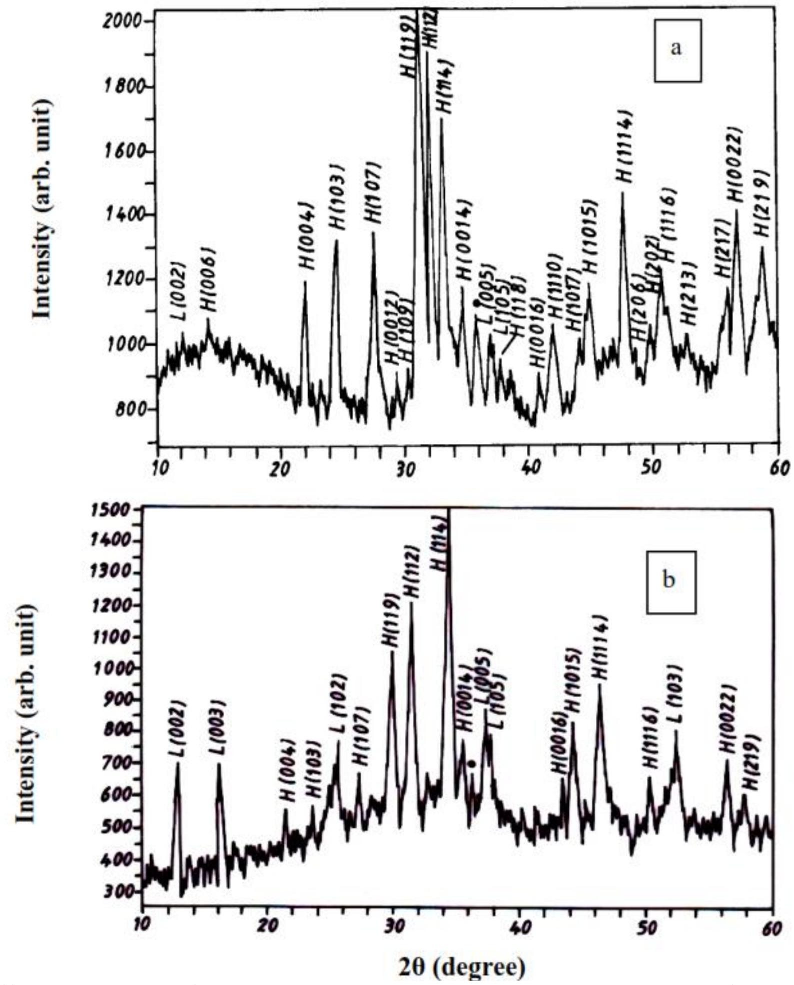

FIGURE 5: X-ray diffraction patterns of $Tl_{1.6}Hg_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$ samples sintered at 860°C for a) 3h and b)6h in air. H-HighT$_c$ phase, L-low T$_c$ phase-impurity phase ($Sr_2Ca_2Cu_7O_8$).

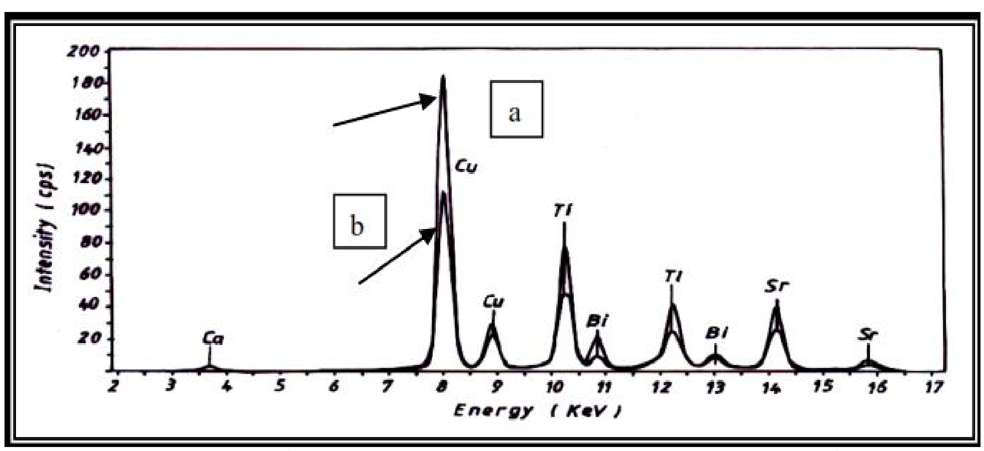

FIGURE 6: XRF patterns of the sample $Tl_{1.8}Bi_{0.2}Sr_2Ca_2Cu_3O_{10+\delta}$ sintered at 860C for (a) 3h (b) 6h.

TABLE 1: Variation in T$_c$ values, lattice parameters and oxygen content with different sintering times

Samples	T$_s$(°C)	t$_s$ (h)	a (Å)	c (Å)	T$_C$(K)	δ
$Tl_{1.6}Hg_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$	860	3	3.721	37.616	120	0.298
	860	6	4.122	36.611	100	0.127
$Tl_{1.8}Bi_{0.2}Sr_2Ca_2Cu_3O_{10+\delta}$	860	3	4.125	31.232	100	0.158
	860	6	4.330	30.872	80	-0.293

CONCLUSION

The outcome foundation of above data is the following:

1. The optimum sintering time which was characteristic for the growth of high-T_c phase equal to 3h.
2. Cuprates with a Hg containing have a higher T_c than those Bi containing.
3. Prolonging the sintering time to 6h lead to decrease the transition temperature.
4. XRD pattern analyses have shown a tetragonal structure, and there were at least two superconducting phases.

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