Enhancement of Superconducting Critical Temperature in Bi(Pb)-Sr-Ca-Cu-O System by Li-doping

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Abstract

We have studied the superconducting transition of the high-T_c Li-doped Bi(Pb)-Sr-Ca-Cu-O superconductors by the DC-resistivity and AC-susceptibility measurements. It was found that Li^+ cations are partially substituted for Cu^{2+} ions. Doping hole by Lithium substitution was supposed to take place in both OP and IP CuO_2 planes. Consequently, the hole concentration increases in the CuO_2 planes. The onset temperature of superconducting transition, $T_{c, onset}$ was observed to increase with Li-doping content as well as the sintering time at 850ºC. We suppose that the optimum hole doping was obtained at 5% Li-doping and the sintering period of 20 days (S05B) with the value of $T_{c, onset} > 116$ K.

Keywords: High-T_c superconductivity, Li-doping, Bi-2223, Bi-2212

1. Introduction

One of the typical high-T_c cuprates is Bi-based superconducting system. The high-T_c superconductors of the Bi–Sr–Ca–Cu–O (BSCCO) system were discovered by Maeda et al. in 1988 [1]. The composition of these materials is determined as $Bi_2Sr_2Ca_{n-1}Cu_nO_{4+2n+\delta}$ with n being 1, 2, and 3. These compounds are distinguished as Bi-2201 ($n = 1$), Bi-2212 ($n = 2$) and Bi-2223 ($n = 3$), where T_c of Bi-2201, Bi2212 and Bi2223 are 20 and 90, 110 K, respectively. The number of the CuO_2 planes increases with increasing n. In bilayer Bi-2212, two CuO_2 planes homogeneous. However, in trilayer Bi-2223, two inequivalent CuO_2 planes, that is, the outer CuO_2 planes (denoted as OP) with a pyramidal (five) oxygen coordination and the inner planes (IP) with a square (four) oxygen coordination. It might be that the outer layers supply a sufficient density of holes, while the inner layers provide a place for strong pairing correlation, both working cooperatively to enhance T_c [2]. Here, one of the main factors influences on the high-Tc superconductivity of Bi-based high-T_c superconductors is also the hole concentration of the CuO_2 plane. The doping hole concentration could be changed by the oxygen content, the cation substitution in the "blocking layer", and especially the substitution of Cu^{2+} by the suitable ones. The doping is varied by changing the oxygen content of the sample [3] and the partially Y^{3+} substitution for Ca [4, 5]. The experimental results of the appearance of coherence intensity at Fermi level were explained by the shift of the chemical potential to the top of the valence band combined with the shift of spectral weight from high- to low energy states. The change of the Cu-O-Cu bonding angle was observed affecting on the metal-insulator transition. The interface high-T_c superconductivity can even be occurred within a single CuO_2 plane [6]. In the other hand, apical oxygen ordering seems to be very important factor that govern strongly on the high-T_c superconductivity [7]. By minimizing Sr site disorder at the expense of Ca site disorder, the author demonstrates that the T_c of $Bi_2Sr_2CaCu_2O_{8+\delta}$ can be increased to 96 K cation disorder at the Sr crystallographic site is inherent in these materials and strongly affects the value of T_c [8]. A new T_c record of 98 K can be attained in Bi-2212 superconductor by reducing Bi content at Sr sites as much as possible [9]. According to M. Qvarford et al., Bi-O layers are essential for the doping of the CuO_2 layers in $Bi_2Sr_2CaCu_2O_8$ [10].

Furthermore, the properties of cuprate layers in Bi-2223 are distinct physical properties. By using NMR method, B.W. Statt showed that the transferring of charge from the bismuth layer (charge reservoir) to the middle CuO_2 layer is partially screened by sandwiching CuO_2 layers, there was lower hole concentration in that layer and enhancing the antiferromagnetic spin fluctuations [11]. Recently, the band splitting in the optimally doped trilayer $Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$ was observed by using ARPES spectroscopy. They made a distinction the energy gap of middle CuO_2 plane (IP) at underdoped region and outer planes (OP) in overdoped region are 60 meV and 43 meV, respectively [12]. Furthermore, the T_c is proportional to superconducting

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energy gap and hole concentration. Nonetheless, due to the strong phase fluctuations in the underdoped IP planes, T_c may be reduced compared to the large pairing amplitude of IP. Kivelson examined a system with alternating two CuO_2 planes as a model of multilayer cuprates; one plane has a large superconducting gap but a very low superfluid density, and the other one has a very small superconducting gap but a high superfluid density. The result shows that phase stiffness of the low-superfluid-density plane is increased through coupling with the high-superfluid density plane, which causes the enhancement of superconducting gap and T_c [13, 14]. Some latest results of ARPES in Bi-2212 were given by Y. He and Co-authors: the bosonic coupling strength rapidly increases from the overdoped Fermi liquid regime to the optimally doped strange metal [15]. The strength of Cooper pairing determined by the unusual electronic excitations of the normal state. Therefore, electron-boson interactions are responsible for superconductivity in the cuprates [16].

With approximate ionic radii, cations Li^+ (0.68 Å) were supposed to be substituted for Cu^{2+} (0.72 Å) ones. Kawai at al. [17] first studied the effects of substituting alkaline metals in Bi-2212 compounds. They found that alkaline elements drastically decrease the formation temperature of the Bi-2212 phase. Especially, the critical transition temperature (T_c) was observed to increase by Li- and Na-doping. The doping of Li is effective to raise T_c for both the 2212 phase and the 2223 phase [18]. The liquid phase formed at lower temperature in Li-doped materials promotes the formation and growth of Bi-2212 phase [19-21] and Bi-2223 phase [22-25]. Study of micro-structural characterization of Li-doped Bi-2212 samples in comparison to undoped one, S. Wu and his co-authors [19] reported that Li partially substituted for Cu in the Bi-2212 structure, with possibility of some interstitial Li remaining as well. A change in the lattice parameters of the Bi-2212 phase due to Li-doping was not found. In contrast, c lattice parameter was reported to be increased with increasing Li-doping content [21]. Addition of other alkaline elements like Na, and K to Bi-based superconductors was found to be effective in forming the high-T_c Bi-2212 phase as well as Bi-2223 one [17, 26, 27]. Because of different preparation conditions and starting chemical composition, Lithium may be substituted for copper at certain content. Therefore, it is rather difficult to estimate the effect of Li-doping on the high-T_c superconductivity. On the other hand, the superconducting transition temperature of Bi-2223 samples depends on the volume ratio of the superconducting phases (Bi-2223/Bi-2212). The suitable heat regime is needed to form and growth the superconducting Bi-2223 crystallites from the low-T_c ones like Bi-2201 and Bi-2212 [22-25]. In addition to partially substitution for Cu^{2+}, Li^+ cations can either combine in none-superconducting matrix in which Bi-2223 grains are embedded or reside as defects in Bi-2223 superconducting grains [22]. These defects were assigned as magnetic pinning centers which influence on the microstructure as well as the critical current density of the superconducting Bi-2223 material.

In this paper, we report some new results in the enhancement of high-T_c superconductivity of Li-doped Bi(Pb)-Sr-Ca-Cu-O superconductors.

2. Experimental

Four samples were prepared by solid-state reaction method. Starting from high impurity Bi_2O_3, PbO, CuO oxides and $SrCO_3$, $CaCO_3$ and Li_2CO_3 carbonates (3N-4N); these were weighed and mixed following the nominal compositions of $Bi_{1.6}Pb_{0.4}Sr_2Ca_2(Cu_{1-x}Li_x)_3O_{10+\delta}$ (with x = 0.0, 0.05, and 0.15). The corresponding powders were calcined at 800°C for 24 h with some additional annealing and grinding steps. Then, three samples were sintered at 850°C for 10 days: S00A (x=0.00), S05A (x=0.05) and S15A (x=0.15). The fourth sample S05B was lasted for a double period (20 days) of sintering at the same temperature of 850°C. Identification of phases that exists in the samples was done by using Siemens X-ray diffractometer D8 with Cu-K$_\alpha$ radiation (λ=1.5406 Å) in the range of 2θ = 20-60°. Specimens were shaped in square bar with their dimensions of $2\times2\times12$ mm^3 and attached to the cold finger of a Helium closed-cycle system (CTI Cryogenic 8200) where they were cooling down and heating up in the temperature range of 20-300 K. The DC-resistivity are measured using four-probes technique with the constant DC current of 10 mA. AC-susceptibility were performed using lock-in amplifier techniques, in AC field amplitude of 2 A/m at frequency of 1 kHz.

3. Results and discussion

3.1 X-ray powder diffraction

Fig. 1 shows X-ray diffraction patterns of four superconducting samples: S00A, S05A, S15A, and S05B. As can be seen, all three samples consist of a mixture of Bi-2223, and Bi-2212 phases as the major constituents. In this measurement, it is hardly to recognize the existence of Bi-2201 phase. Almost intensities of the Bragg reflection peaks of the second phase Bi-2212 increase with increasing Li-doping content from undoped sample S00A (x=0.00) to S05A, and obtained the maxima at highest doped sample S15A (x=0.15). In addition, the CuO phase (*) can be detected at a small amount. The crystal structure of Bi-2223 phase is pseudo-tetragonal unit cell (I4/mmm). The crystal lattices of undoped sample are c = 37.109 Å and $a \sim b$ = 5.402 Å. Some

very small changes of these lattices by Li-doping were observed. The data were given in Table 1.

The volume fractions of the phases can be estimated using various methods. We can use all peaks of the Bi-2223, Bi-2212 and Bi-2201 phases for estimation of the volume fractions of the phases, respectively (see more detailed in reference [28]).

Table 1. Lattice parameters and volume fractions of four Li-doped Bi-2223 samples.

Sample	Volume fraction		Lattice	Parameters
	%Bi-2223	%Bi-212	a(Å)	c(Å)
S00A	80	20	5.4020	37.109
S05A	75	25	5.4025	37.141
S15A	65	35	5.4027	37.108
S00B	77	23	5.4027	37.112
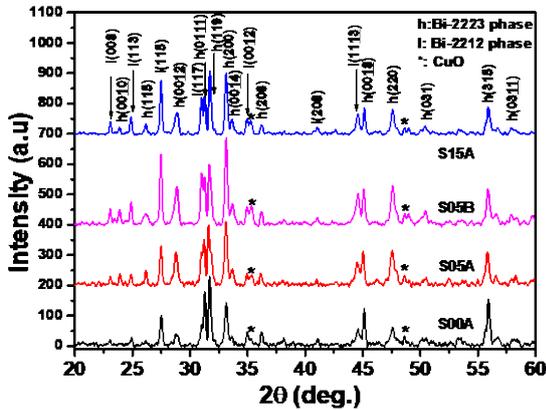
Fig. 1. X-ray diffraction patterns of four Li-doped superconducting Bi-2223 samples; (hkl): Miller indices of the crystal planes belong to Bi-2223, Bi-2212 phases, and major impurity phase is CuO (*).

Here, we only used all the peaks of the two mentioned phases Bi-2223 and Bi-2212 for the characterization of the phase formation of the samples and ignore the voids, namely:

$$Bi-2223(\%) \approx \frac{\sum I(Bi-2223)}{\left[\sum I(Bi-2223)+\sum I(Bi-2212)\right]} \quad (1)$$

Where, I is the intensity of the present phases.

The results show that volume fraction of Bi-2212 phase increases from 20% (sample S00A) to 25% (sample S05A) and obtain maximum value 35% for S15A sample. Inversely, the volume fraction of Bi-2223 phase decreases from S00A (80%) to S05A (75%) and S15A sample (65%). In the small doping (x=0.05), when we last the sintering time up to 20

days (for S05B sample), the volume Bi-2223 phase fraction slightly increases to 77% in comparison with 75% of S05A sample (see more on Table 1).

3.2 DC-resistivity

The dc-resistivity characterization of all four samples was depicted in Fig.2. The temperature derivative of $\rho(T)$ curves was given in Fig.3. The resistivity curves $\rho(T)$ of the four Li-doped Bi-2223 samples were depicted in Fig.2. In the normal state (120-300 K), the characterization of the un-doped sample (S00A) as well as the others is approximately proportional to the temperature. In the guide-to-eyes definition, the superconducting temperature T_c seems to be larger than 110 K. However, the resistivity only can reach zero at lower temperature.

Table 2. The values of resistivity at 300K and relative resistivity of four Li-doped Bi-2223 samples determined from resistivity curves in Fig. 2.

Samples	$\rho(300K)$ (mΩ.cm)	$\rho(120K)/$ $\rho(300K)$	$\rho(T_{c,onset})/$ $\rho(300K)$
S00A	8.53	0.282	0.193
S05A	11.90	0.305	0.234
S15A	11.30	0.290	0.255
S05B	5.68	0.278	0.248
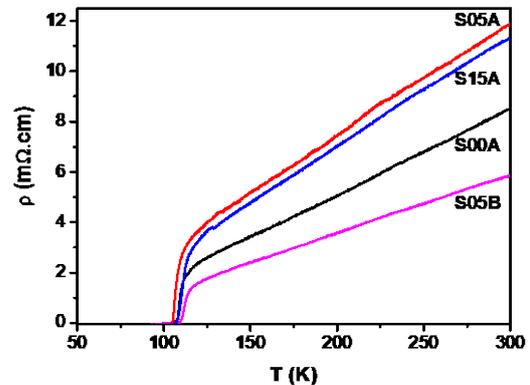
Fig. 2. Resistivity vs. temperature curves of four Li-doped superconducting Bi-2223 samples.

For x=0.00, The resistivity at 300K, $\rho(300K)$, is equal to 8.53 mΩ.cm. It increases with increasing the Li-doping content up to 11.9 mΩ.cm (for S05A). Approximately, it increases about 40%. At highest Li-doping content, the resistivity of S015 sample is a little smaller than that of S05A sample (see more in Fig. 2). However, when the sintering time was double (20 days) the resistivity of S05B was drastically

reduced to a half (5.68 mΩ.cm) in comparison with the value of S05A sample (the detailed values given in Table 2). At the same heating time, the metallic behavior of the different Li-doping level can be estimated by the relative resistivity $\rho(120K)/\rho(300K)$ and $\rho(T_{c,onset})/\rho(300K)$. The metallic behavior seems to decrease with increasing Li-doing content. This influence can be explained by the partially substitution of Li^+ for Cu^{2+} in the CuO_2 plane. We can assign the starting point of temperature at which resistivity begins dropping, $T_{c,onset}$. In contrary, $T_{c,0}$ is the temperature where the resistivity totally becomes zero. In the middle, the critical temperature can be measured at the temperature of the peak point of different resistivity curve, T_c. The critical parameters were given in Table 3.

Table 3. The critical temperatures and the transition width of four Li-doped Bi-2223 samples determined from differential resistivity curves in Fig. 3.

Samples	$T_{c,0}(K)$	T_c (K)	$T_{c, onset}$ (K)	ΔT_c
S00A	107.2	108.7	111.2	4.0
S05A	105.0	107.0	110.8	5.8
S15A	105.6	110.5	116.0	11.4
S05B	108.5	111.6	116.5	8.0
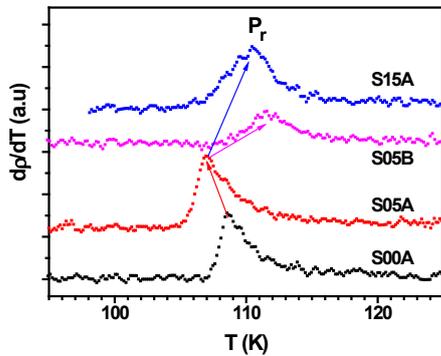
Fig. 3. Differential Resistivity vs. temperature curves of the superconducting Li-doped Bi-2223 samples. For clarifying, we have added up the curves with a certain value.

The shift of the differential peak P_r (fixed at T_c) to the higher position at higher Li-doping (S15A) as well as longer period of sintering (S05B) suggesting us about the optimum doped high-T_c superconducting phase of Bi-2223. Obviously, the substantial volume fraction of this new high-T_c superconducting phase was obtained in S05B sample. As a result, the superconducting transition become sharper.

3.3 AC-susceptibility

Fig. 4 shows the temperature dependent AC-susceptibility of un-doped Bi-2223 sample (S00A). The diamagnetic onset temperature is approximately 111.3K ($T_{c,D}$). This is the temperature at which the real part (χ') starts dropping as well as the imaginary part (χ'') turning up. At this temperature point, AC field ($H_{ac}=2A/m$) is high enough to penetrate the grains. The flux is gradually driven out of the inter-granular volume when the temperature decreases up to $T_{ID}=101K$ for the measurement ($H_{ac}=2A/m$, $f=1kHz$). At this temperature, the whole volume of the sample expected to be shielded by the super-current circulating in the sample and hence the diamagnetic signal becomes saturation (full Meissner effect).
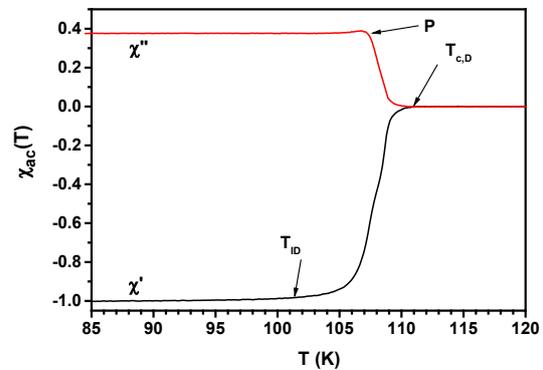
Fig. 4. Temperature dependent ac-susceptibility of un-doped superconducting Bi-2223 sample in AC magnetic field of 2A/m at frequency of 1kHz.
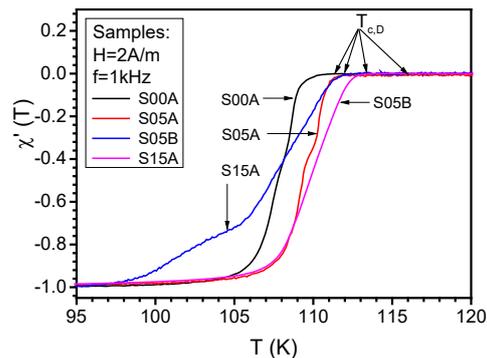
Fig. 5. Temperature dependence of real parts (χ') of AC-susceptibility curves of four Li-doped superconducting Bi-2223 samples.

For clarify, we draw graphs of real parts (χ') and imaginary parts (χ'') in separated Fig.s 5 & 6, respectively. As above results, the diamagnetic onset

temperature ($T_{c,D}$) of the un-doped sample equal to 111.3K. This critical value is approximately to the $T_{c,onset}$ determined from the resistivity curve (111.2K). But, at low Li-doping content ($x=0.05$), the diamagnetic onset temperature, $T_{c,D}$ increases to 111.9K in contrary to the decrease of $T_{c,onset}$ (110.8K). We suppose that Li^+ cations can substitute for Cu^{2+} ones as well as create some defects in the Bi-2223 crystallites. Because the AC-susceptibility can measure the Meissner signal of volume fraction of Bi-2223. However, the onset of critical transition happens at the point of superconducting coherence of the sample. By further Li-doping content ($x=0.15$), $T_{c,D}$ increases to the value larger than 113.4K. It is a bit rather difficult to determine the diamagnetic onset temperature exactly because of the signal interference.

Table 4. The values of the diamagnetic onset ($T_{c,D}$), ideal diamagnetic (T_{ID}) and loss peak temperatures (T_p) obtained from AC-magnetic susceptibility measurements (**Fig.s 4, 5 &6**).

Samples	$T_{c,D}$ (K)	T_{ID} (K)	$\Delta T_{c,D}$ (K)
S00A	111.3	101	10.3
S05A	111.9	103.5	7.8
S15A	> 113.4*	96.0	17.2
S05B*	116.2	104.5	11.7
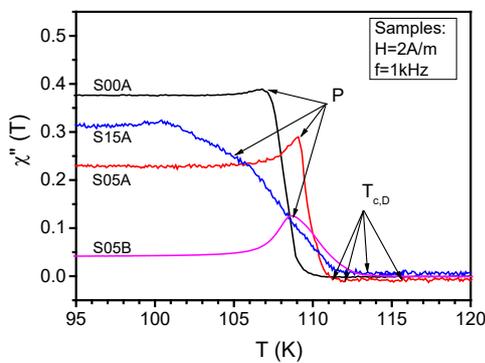
Fig. 6. Temperature dependence of imaginary parts (χ") of AC-susceptibility curves of four Li-doped superconducting Bi-2223 samples.

When the sintering time was last for 20 days, $T_{c,D}$ can reach to higher temperature (116.2K) even the Li-doping is low (S05B). The increasing tendency $T_{c,D}$ is similar to that of $T_{c,onset}$ taken from resistivity measurements. The full Meissner effect (T_{ID}) appears at lower temperature in comparison with the zero-resistivity temperature ($T_{c,0}$). This difference can be explained the particular weak-link behavior of Bi-

based superconducting materials (see more in Tab. 4). Additionally, the loss peak (P) was very much broaden for higher doping content (S15A), and the intensity was dramatically reduced in longer sintering period (S05B). The broaden of superconducting transition in sample S15A can be explained by the different in hole concentration as well as the effect of higher volume content of Bi-2212 phase.

3.4 Discussion

For all studied samples, there always exist two major superconducting phases Bi-2212 and Bi-2223. At the same sintering period, the higher Li^+ cations we doped, the larger volume ratio of Bi-2212 phase we've got. This is due to relatively preferable of Li^+ ions in Bi-2212 phase [29]. In the other hand, the growth of crystallites Bi-2223 phase taken place by inserting extra Ca/CuO_2 plane in the Bi-2212 matrix. This crystal growth was governed by the microscopic kinetics and diffusion mechanism [30]. Li^+ cations have been substituted partly for Cu^{2+} ions in CuO_2 planes of the superconducting phase Bi-2223. The Lithium substitution affects the quality of the sample on many aspects. The high-T_c superconducting onset ($T_{c,onset};$ $T_{c,D}$), and the transition temperature range increase with increasing Li-doping. Li^+ cations of the liquid phase can diffuse into the superconducting grain from the boundary at the same times with their growth. The little mismatch of Li^+ in comparison with Cu^{2+} may restrain the growth of Bi-2223 phase from the Bi-2212, as well as that of Bi-2212. However, with quite a long time of 10 days sintering at 850°C, the existence of Bi-2201 phase could be very small, and enough condition for the formation of Bi-2212 phase with high volume fraction. It is supposed that Bi-2212 phase is situated at grain boundary of Bi-2223 phase [31].

The diffusion of Li^+ cations into the superconducting grain following two aspects: they can substitute for Cu^{2+} on CuO_2 planes as well as make defects called as intra grain defects which can decrease the superconducting volume of the grain. Because of the different sizes of the superconducting grains, the doping level owns a wide range. Therefore, the hole concentration in CuO_2 planes are also different from grain to grain. As a result, the superconducting transition broaden with the Li-doping (for sample S15A). It was found that the Bi-2212 phase on the grain boundaries is likely to play the role of weak links and consequently reduces the inter-granular coupling [28]. For S05B sample, with quite a long time of sintering (20 days at 850°C) the optimum hole doping we have got with the shaper superconducting transition. The starting temperatures of superconducting transition at 116 K for S15A

sample, and 116.5 K for S05B sample are quite larger than that of un-doped sample (111.3K). As a result, we suggested that the Li-doping make appearance of optimum doped high-T_c superconducting phase of Bi-2223. There are some reasons for explaining the higher superconducting transition in Li-doping:

a) Li^+ cations partially substituted for Cu^{2+} ones in both the outer planes (OP) and inner CuO_2 planes (IP) of Bi-2223 phase. Nevertheless, the substitution Li^+/Cu^{2+} taken place with the growth of Bi-2223 crystal grains at the same time. At first, Li^+ substituted for Cu^{2+} cations in the outer planes of both Bi-2212 and Bi-2223 phase. This increases the hole concentration at different levels. Therefore, the superconducting transition extended in a large range of temperature. Then, the optimum hole doping is amongst of those levels.

b) When the sintering time was raised up to twice (for sample S05B). The longer sintering time we took the more chance Li^+ ions be substituted for Cu^{2+} cations, especially in IP planes. The increase of volume ratio of optimum doped high-T_c phase of Bi-2223 are explained by the adjustment of the ratio Ca^{2+}/Sr^{2+} [8], the decrease of Bi^{3+} at Sr^{2+} sites [9], or the ordering of apical oxygen [7, 32]. In addition, the normal resistivity decrease, and the weak links improve. In this work, Li-doping increase the superconducting critical temperature at quite high values (4-5 K) in comparison with that of Bi-2223 whiskers (1.2 K) or ceramic superconducting compounds [29, 33]. Here, doping hole by lithium substitution was supposed to take place in both OP and IP CuO_2 planes.

The substitution of other elements for copper have been taken by some groups in references [34-38]. The depression of T_c was observed for Bi-2223 materials with the dopants of 3d-metals like Ni, Co [34, 35]. The positive effect of the high-T_c superconducting transition temperature have not been observed by 4f-element doping [36-37]. Even though in the same group as Li element, Na also do not exhibit the positive signal of high-T_c superconductivity [38].

4. Conclusion

We have investigated the enhancement of high-T_c superconductivity in Li-doped Bi(Pb)-Sr-Ca-Cu-O superconductors by both DC-resistivity and AC-susceptibility measurements. Doping hole by Lithium substitution for Copper was supposed to take place in both OP and IP CuO_2 planes. The onset temperature of superconducting transition, $T_{c, onset}$ was observed to increase with Li-doping content as well as the sintering time at 850°C. In this work, the optimum

hole doping was obtained at 5% Li-doping and the sintering period of 20 days (S05B) with the value of $T_{c, onset} > 116$ K. This transition value is approximately 5K larger than the one observed from un-doped sample (S00A).

Acknowledgment

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