

# Effects of Li Substitution in Bi-2223 Superconductors

O. Bilgili · Y. Selamet · K. Kocabaş

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**Abstract** The effects of Li substitution on the properties of high temperature superconductor  $\text{Bi}_{17}\text{Pb}_{0.3}\text{Sr}_2\text{C}_2\text{Cu}_{3-x}\text{Li}_x\text{O}_y$  were investigated. The samples were prepared by substituting Li ( $x = 0.00\text{--}0.20$ ) with changing ratios by a solid state reaction method. The samples were characterized by X-ray diffraction, DC electrical resistivity, AC magnetic susceptibility, and scanning electronic microscopy (SEM). The X-ray diffraction studies were done at room temperature and the lattice constants of the material were determined by indexing all the peaks observed.

This study shows that there are two coexisting phases; high- $T_c$  (2223) phase and low- $T_c$  (2212) phase. The lattice structure of the material belongs to the orthorhombic unit cell. The volume fraction was estimated from the intensities of Bi-(2223) and Bi-(2212) phases. The sample with 20 wt% of added Li showed the higher volume fraction of Bi-(2223) phase formed (81%) compared to the other samples. The DC electrical resistivity of all the samples decreased as the wt% of Li increased. Both the onset critical temperatures  $T_c$  (onset) and zero electrical resistivity critical temperatures  $T_c$  ( $R = 0$ ) of the samples were determined from the DC electrical resistivity measurements. The observed value of the onset critical  $T_c$  (onset) temperature was 110 K agreeing well with the magnetic susceptibility measurements. We obtained  $T_c$  onset at 112 K from AC magnetic susceptibility measurements.

**Keywords** Bi-based superconductors · Li substitution · AC susceptibility · Scanning electronic microscopy · X-ray diffraction

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## 1 Introduction

A tremendous amount of work concerning the preparation, the superconducting properties, and the structure of these compounds has been done since the discovery of high- $T_c$  superconductivity in the Bi–Sr–Ca–Cu–O (BSCCO) system [1]. It is now well known that the Bi-based system has layered structure and according to their compositions, it has three different phases which are called Bi-(2201), Bi-(2212), and Bi-(2223) [2].

In the general formula of  $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_{2n+4+y}$ ,  $n$  is 1, 2, or 3 and it refers to the number of  $\text{CuO}_2$  layers which yields the 10, 95, or 110 K superconducting phases, respectively. Hence, the Bi-(2201), Bi-(2212), and Bi-(2223) phases have single, double, and triple layers of  $\text{CuO}_2$  in the sub-unit cell, respectively, and more planes are believed to be associated with higher value of  $T_c$  ( $R = 0$ ) [3].

It is very difficult to prepare the Bi-(2223) phase, which has the highest  $T_c$  among the family, in a single phase. The superconducting properties of  $\text{BiSrCaCuO}$  superconductor can be controlled by the addition or substitution of the elements with different ionic radius and different bonding characters. The enhancement or destruction of the superconducting properties depends on the characteristics of the dopant in the crystal structure. Therefore, most of the research efforts to improve the (2223) phase formation of this system were concentrated on the substitution studies [4]. Pb is the

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most important substituting element that influences the microstructure, phase composition, and the related superconducting properties of the BSCCO system. The presence of Pb (usually in the form of PbO) in the initial mixture favors the reaction kinetics of the 110 K- $T_c$  (2223) phase [5].

There have been great improvements in enhancing transport properties of this high- $T_c$  superconductor since the discovery of (Bi,Pb) $_2$ Sr $_2$ Ca $_2$ Cu $_3$ O $_y$  (Bi-2223) superconductor. However, the major limitations of the Bi-2223 system superconductor applications are the intergrain weak links and weak flux pinning capability. It was shown that one of the major current limiting factors for Bi-2223 is the presence of residual secondary phases [6].

Alkaline metals (Li, Na, K, Rb, Cs) could be the candidate for the substitution since their ionic radii (73–181 pm) overlap those of Bi, Pb, Sr, Ca, and Cu. The ionic radii of Cu and Li are 0.72 and 0.76 Å, respectively. Moreover, alkaline metals have a +1 valence state; hence, their addition is attractive from the point of changing carrier concentrations [7]. The effects of substituting alkaline metals for Bi, Sr, Ca, and Cu in Bi $_2$ Sr $_2$ CaCu $_2$ O $_x$  were studied by [7, 8]. In these works, the  $T_c$  was increased by Li and Na doping, but was decreased by K and Rb doping. It was also found that alkaline metals drastically decreased the formation temperature of the Bi-2212 phase.

The resistivity drop in temperature dependent measurements usually characterizes the transition from a normal to the superconducting state. However, the magnetic manifestation of zero resistivity is that a material is a superconductor if it exhibits perfect diamagnetic shielding. That is, its susceptibility  $\chi$  is exactly  $-1$  (in SI units, where numerical results must be corrected for any sample demagnetization factor). Therefore, an AC susceptibility measurement can be used to determine the critical temperature  $T_c$  [9].

The systematic studies on the LiF and LiCl doped Bi-2223 systems revealed that a small quantity of lithium compound helped the formation of the high- $T_c$  superconducting phase (Bi 2223) [10, 11]. However, this induces the formation of the low- $T_c$  superconducting phase (Bi-2212) at higher doping levels. The critical temperature  $T_c$  of the 110 K superconducting phase Bi-2223 reaches 119.5 K for oxygen mole fraction  $y = 0.15$  in the case LiF doping and 112.5 K for  $y = 0.1$  in the case of LiCl addition [12].

In this work, we investigated the properties of Bi $_{1.7}$ Pb $_{0.3}$ Sr $_2$ Ca $_2$ Cu $_{3-x}$ Li $_x$ O $_y$  ( $x = 0.00, 0.05, 0.10, 0.20$ ) specimens where Li was added in the form of Li $_2$ O. We reported measurements of electrical resistivity and magnetic susceptibility as a function of temperature and intergrain properties of Bi-2223 superconductors by using SEM photographs. We also reported XRD measurements in order to calculate the lattice parameter and the relative portion of Bi-2223 and that of Bi-2212 phases.

## 2 Experimental Details

Samples of nominal composition Bi $_{1.7}$ Pb $_{0.3}$ Sr $_2$ Ca $_2$ Cu $_{3-x}$ Li $_x$ O $_y$  ( $x = 0.00, 0.05, 0.10, 0.20$ ) were prepared by the conventional solid-state reaction method. The starting powders were corresponding stoichiometric quantities of high purity (99.99%) Bi $_2$ O $_3$ , PbO, SrCO $_3$ , CaCO $_3$ , CuO, and Li $_2$ O. The powders were weighted in necessary atomic ratios. The substitution ratios varied to have  $x = 0.00$  to  $x = 0.20$  resulting in five groups of samples prepared with each dopant labeled as A ( $x = 0.00$ ), B ( $x = 0.05$ ), C ( $x = 0.10$ ), D ( $x = 0.15$ ), and E ( $x = 0.20$ ). The weighed powders were thoroughly mixed and grounded by using a mortar and pestle and calcined at 800 °C for 20 h in air. The powder mixture was again calcined at 820 °C for 20 h in air after a second intermediate grinding. The final product after calcinations was grounded once more. Then these powders were pressed under 450 MPa pressure using a press machine (Graseby Specac) to form them into pellets 13 mm in diameter and 3 mm thick. The prepared pellets were finally sintered at 855 °C for 150 h in air.

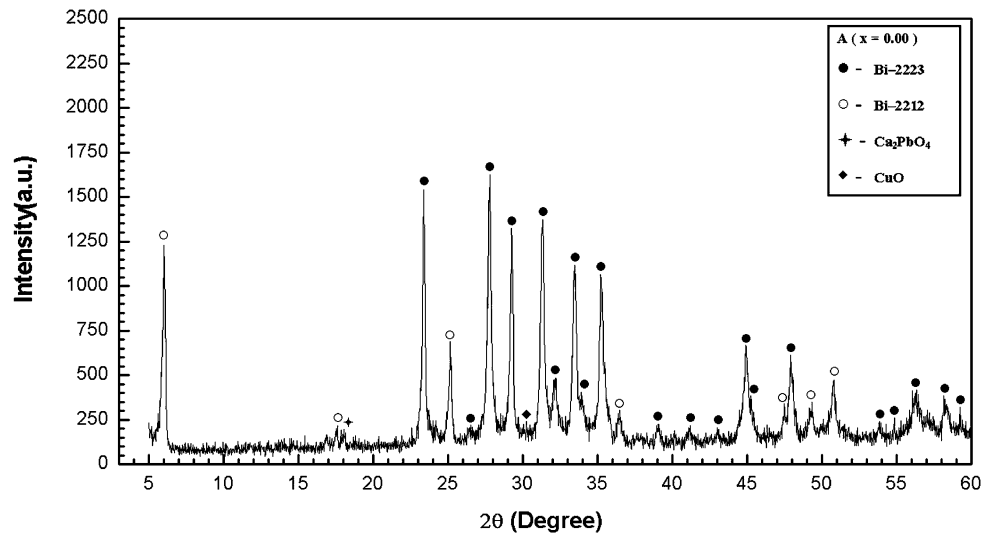
To study the effect of the Li addition on the properties of Bi-based BSCCO superconductor samples were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), resistivity–temperature ( $R$ – $T$ ) measurements, temperature dependent magnetic susceptibility measurements, and volume density measurements. SEM imaging was used to examine the surface morphology and grain structure of the samples. Scanning electron microscopy (SEM) images were taken by using a Phillips XL-30S FEG microscope.

The  $R$ – $T$  behavior of the samples from 30 K to 250 K was investigated in order to determine superconducting transition temperature of the prepared samples. The electrical resistance was measured by the standard four-probe method. The X-ray powder diffraction pattern of each sample was obtained using CuK $_{\alpha}$  radiation in the range  $2\theta = 5 - 60^\circ$  by a Rigaku DMax 2200 Diffractometer. Phase purity and the lattice parameters were obtained from these XRD patterns. The mutual inductance bridge method was used for AC magnetic susceptibility measurements in 80–300 K temperature range.

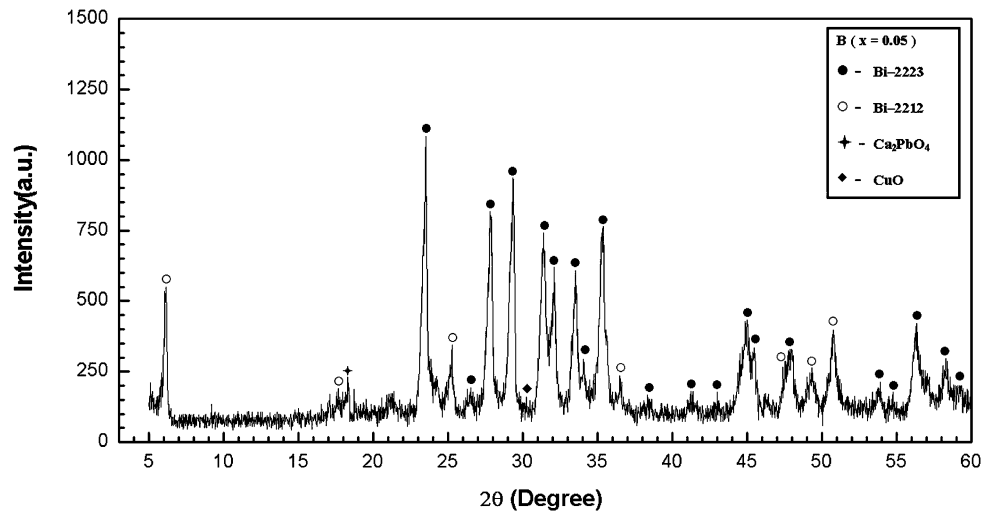
## 3 Results and Discussion

The X-ray diffraction (XRD) patterns of samples Bi $_{1.7}$ Pb $_{0.3}$ Sr $_2$ Ca $_2$ Cu $_{3-x}$ Li $_x$ O $_y$  ( $x = 0.00, 0.05, 0.10, 0.20$ ) indicated that the samples consisted of the low- $T_c$  (2212) and high- $T_c$  (2223) phases and contained small amount of CuO and Ca $_2$ PbO $_4$  (Fig. 1). The CuO peak was identified in all samples at  $2\theta$  values of  $31^\circ$ . A weak characteristic impurity phase Ca $_2$ PbO $_4$  peak at  $2\theta = 17.8^\circ$  was identified in all samples.

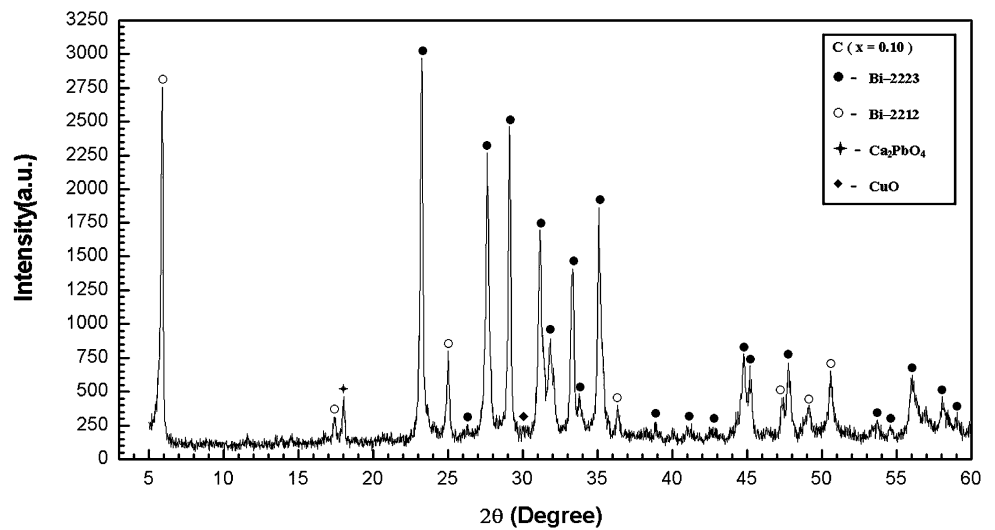
**Fig. 1** XRD patterns of the samples  
 $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_{3-x}\text{Li}_x\text{O}_y$   
 ( $x = 0.00-0.20$ )



(A)

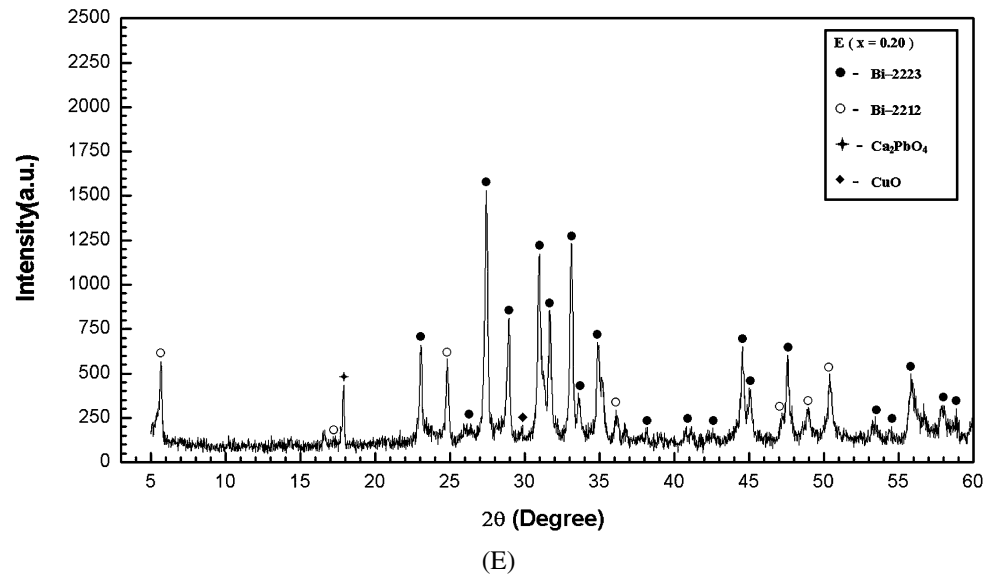
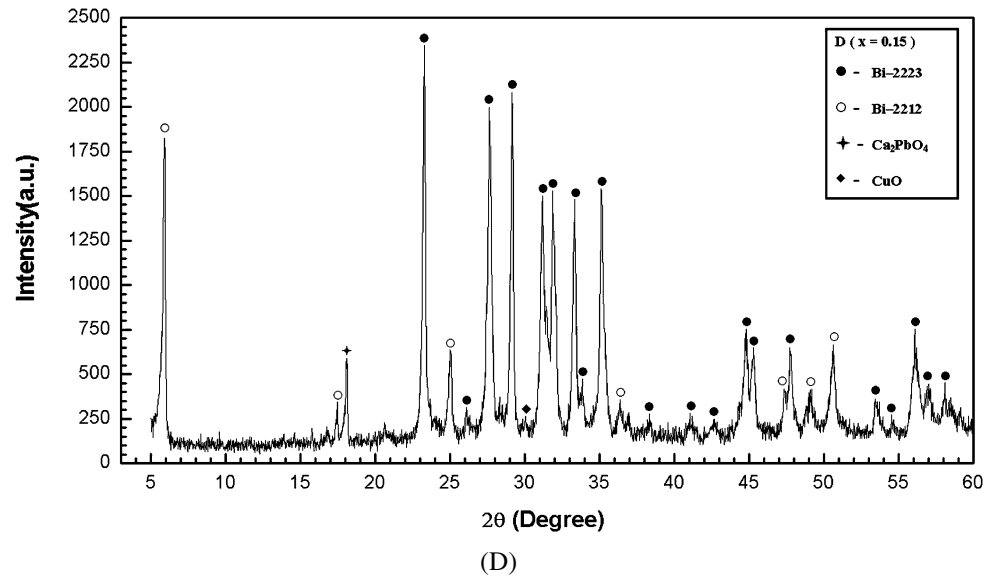


(B)



(C)

Fig. 1 (Continued)



All the samples studied by XRD measurements were mainly consisting of (2212) and (2223) phases, with the (2223) phase being the dominant one. We have used all the XRD peaks of Bi-(2223) and Bi-(2212) phases for the estimation of the volume fraction of the phases using the following formulas:

$$\text{Bi-(2223)\%} = \frac{\sum I(2223)}{\sum I(2223) + I(2212)} \times 100$$

$$\text{Bi-(2212)\%} = \frac{\sum I(2212)}{\sum I(2223) + I(2212)} \times 100$$

where  $I(2223)$  and  $I(2212)$  are the intensities of the ( $hkl$ ) X-ray diffraction peaks for Bi-2223 and Bi-2212 phases, respectively [13]. The calculated relative portion of the samples is listed in Table 1. As seen in the table, samples with

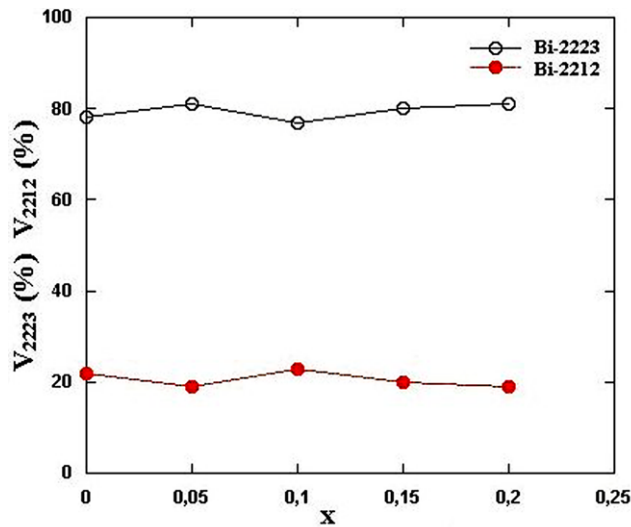
$x = 0.00$  and  $0.05$  contained 78% and 81% of the 2223 phase, respectively. Increasing Li substitution in our samples resulted in a decrease of the intensities of the peaks of the 2212 phase. In samples with  $x = 0.10$ ,  $0.15$ , and  $0.20$  the amount of the 2223 phase was 77%, 80%, and 81%, respectively. The percent amount of the 2223 and 2212 phases as a function of  $x$  evaluated from the XRD patterns of the  $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_{3-x}\text{Li}_x\text{O}_y$  ( $x = 0.00, 0.05, 0.10, 0.20$ ) samples are given Fig. 2.

It was found that the high- $T_c$  (2223) phase has orthorhombic structure with lattice constants  $a = 5.419 \text{ \AA}$ ,  $b = 5.376 \text{ \AA}$  and  $c = 37.06 \text{ \AA}$  [5]. Similar lattice parameters were also observed for compounds in our study. The lattice parameters for the phases were calculated by taking into account the peaks corresponding to Bi-(2223) phases. Unit cell dimension for the 2223 phase of  $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_{3-x}$

**Table 1** Lattice parameters and percentage volume fraction of Bi-(2223) and Bi-(2212) phases in the sample

Sample	Unit cell (Bi-2223 phase)			Volume fraction of phases formed (%)	
	<i>a</i> (Å°)	<i>b</i> (Å°)	<i>c</i> (Å°)	Bi-2223 phase	Bi-2212 phase
A	5.411	5.37	37.069	78	22
B	5.410	5.39	37.073	81	19
C	5.408	5.38	37.079	77	23
D	5.405	5.36	37.092	80	20
E	5.402	5.39	37.095	81	19

Bi<sub>1.7</sub>Pb<sub>0.3</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3-x</sub>Li<sub>x</sub>O<sub>y</sub>  
(*x* = 0.00–0.20)



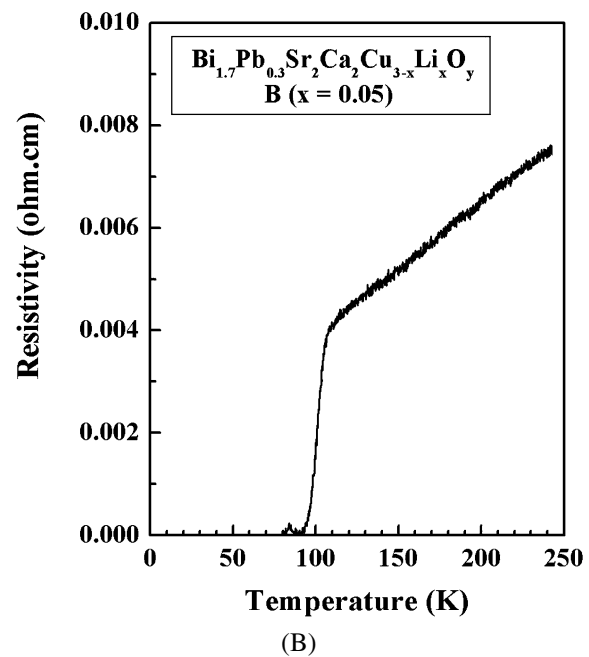
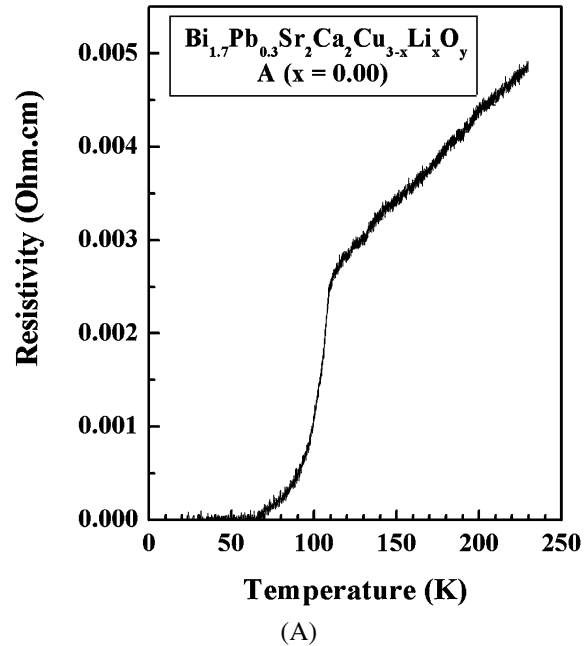
**Fig. 2** Volume fraction of Bi-(2223) and Bi-(2212) phases determined from the X-ray diffraction patterns

Li<sub>*x*</sub>O<sub>*y*</sub> (*x* = 0.00–0.20) samples are given in Table 1. The results obtained in this study were in good agreement with the theoretical results of Bi-2223 system. We observed that the lattice parameters of the samples with Li substitution did not change significantly from the theoretical value.

The electrical resistivity versus temperature plots shows that the resistivity decreases linearly with temperature in the normal state (Fig. 3). All samples displayed a metallic character above onset temperature, which is defined as the temperature where resistance-temperature plot deviates from linearity [14]. It is seen from the plots that the onset temperatures *T<sub>c</sub>* (onset) of the samples are in the close vicinity of 110 K.

The zero resistivity critical temperature *T<sub>c</sub>* (*R* = 0) for the undoped sample (*x* = 0.00) was 70 K. The sample with *x* = 0.20 had the highest critical temperature. The transition temperature width ( $\Delta T_c$ ) of this sample was narrower than those of the other samples.

The critical temperatures *T<sub>c</sub>* (*R* = 0) of Bi<sub>1.7</sub>Pb<sub>0.3</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3-x</sub>Li<sub>*x*</sub>O<sub>*y*</sub> samples with *x* = 0.05, 0.10, 0.15, and 0.20 were 92 K, 96 K, 89 K, and 98 K, respectively, and are tabulated in Table 2. The variation of *T<sub>c</sub>* with Li content, *x* is given in Fig. 4.



**Fig. 3** Temperature dependences of the electrical resistivity for the sample Bi<sub>1.7</sub>Pb<sub>0.3</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3-x</sub>Li<sub>*x*</sub>O<sub>*y*</sub> (*x* = 0.00–0.20)

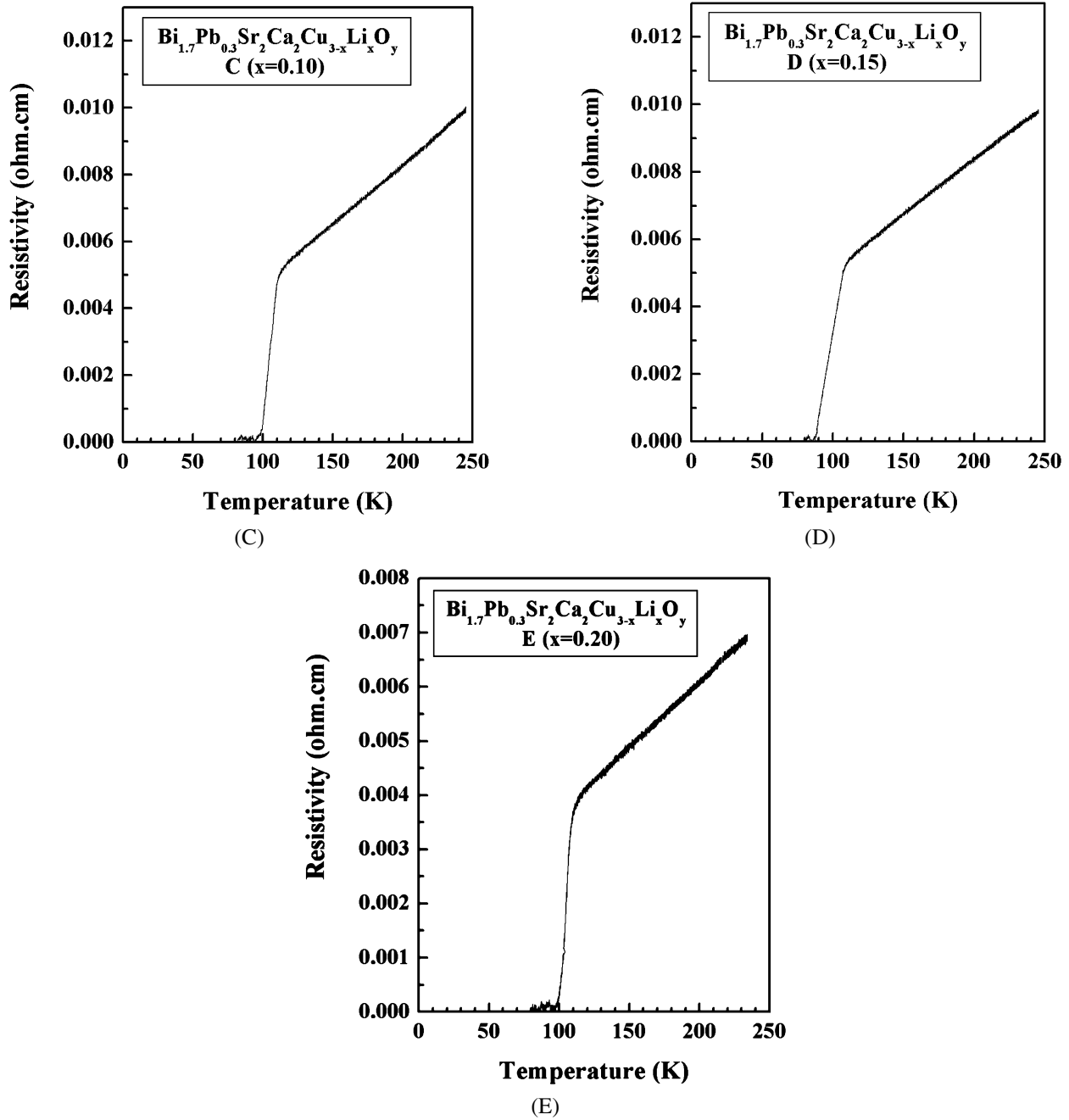
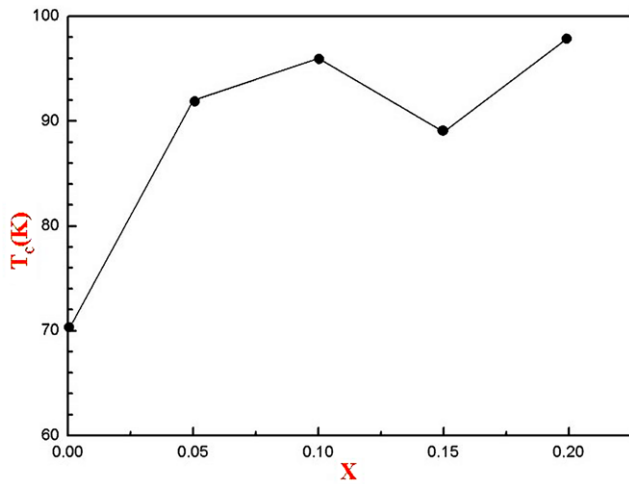


Fig. 3 (Continued)

**Table 2** The critical temperature  $T_{c,zero}$  (K),  $T_{c,onset}$  (K),  $\Delta T_c$  (K) and mass density of the samples  $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_{3-x}\text{Li}_x\text{O}_y$

Sample	Li (x)	$T_c$ (onset) K	$T_c$ (R = 0) K	$\Delta T_c$ K	Mass density ( $\text{g/cm}^{-3}$ )
A	0.00	110	70	40	5.4881
B	0.05	110	92	18	5.4529
C	0.10	110	96	14	5.4377
D	0.15	110	89	21	5.1934
E	0.20	110	98	12	5.1482



**Fig. 4** Variation of critical temperature of Li doped samples of Bi<sub>1.7</sub>Pb<sub>0.3</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3-x</sub>Li<sub>x</sub>O<sub>y</sub> ( $x = 0.00-0.20$ )

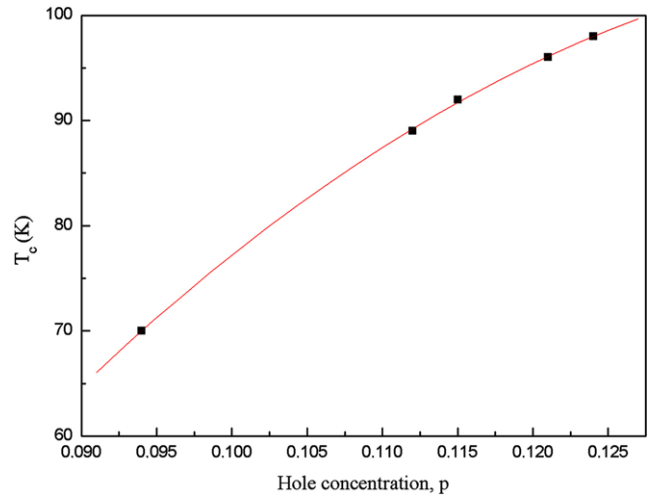
A parabolic relationship holds between the superconducting transition temperature and the hole concentration  $p$ . The carrier concentration  $p$  is calculated by using the relation given below

$$T_c/T_c^{\max} = 1 - 82.6(p - 0.16)^2$$

where  $T_c^{\max}$  is taken as 110 K for the Bi-2223 system. Previous calculations for the unsubstituted Bi-2223 had shown that the values of  $p$  ranged from 0.116 to 0.160. Hence, the  $p$  in the underdoped region can be determined by using  $p = 0.16 - [(1 - T_c/T_c^{\max})/82.6]^{1/2}$  from the measured values of  $T_c$  [15]. Hole carrier concentration increases from 0.094 to 0.124 with increasing Li concentration, shown in Fig. 5. The relation between  $T_c$  and hole concentration is almost parabolic for our samples. The hole concentrations of Bi<sub>1.7</sub>Pb<sub>0.3</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3-x</sub>Li<sub>x</sub>O<sub>y</sub> samples with  $x = 0.00, 0.05, 0.10, 0.15,$  and  $0.20$  were 0.094, 0.115, 0.121, 0.112, 0.124, respectively.

The real part of AC susceptibility,  $\chi'$ , in polycrystalline samples experimentally shows two drops when the temperature is lowered below onset of diamagnetic transition. Correspondingly, the derivative of the  $\chi'(T)$  displays two peaks. The first sharp drop at  $T_c$  is due to the transition within grains. Besides, the second gradual change at  $T_{cJ}$  is due to the occurrence of the superconducting coupling between grains [16].

AC magnetic susceptibility measurements were done for all the samples and almost same behavior of susceptibility versus temperature curves was obtained. The real parts of the AC susceptibility versus temperature measurements of samples are shown in Fig. 6. The diamagnetic transition in the real part of AC susceptibility occurs sharply at  $T_c$  (onset) temperature. The onset temperature of all the samples is  $110 \pm 1$  K. The DC electrical data agree well with AC magnetic susceptibility measurements. When the samples are at



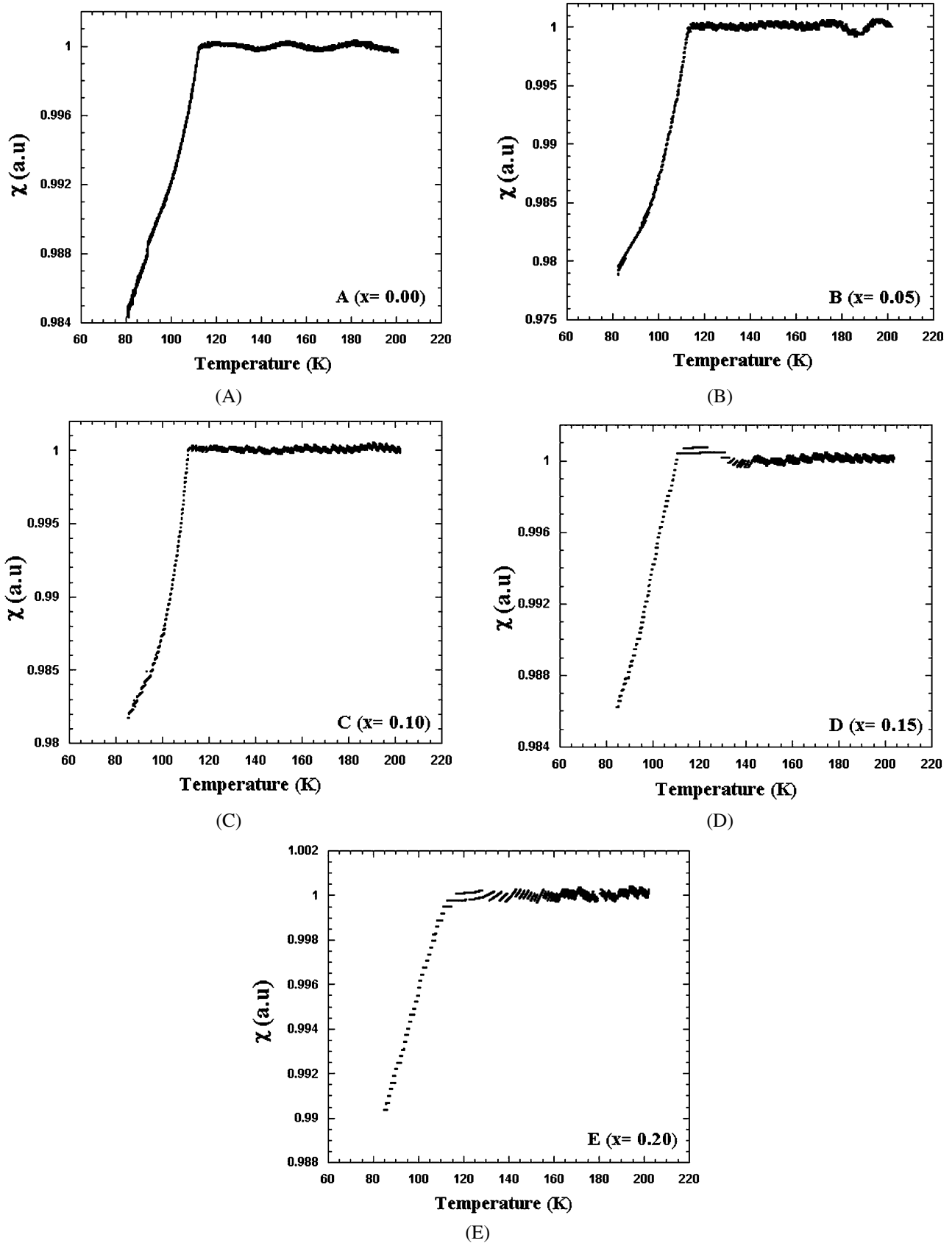
**Fig. 5** Superconducting transition temperature versus hole concentration. Bi<sub>1.7</sub>Pb<sub>0.3</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3-x</sub>Li<sub>x</sub>O<sub>y</sub>. (A)  $x = 0.00$ , (B)  $x = 0.05$ , (C)  $x = 0.10$ , (D)  $x = 0.15$ , and (E)  $x = 0.20$

a temperature just below  $T_c$  ( $R = 0$ ), the superconducting grains first shield the applied magnetic field. This is measured as a negative  $\chi$ .

The SEM images of the samples A, B, C, D, and E are shown in Fig. 7. It is seen that the microstructure consists of large chaotically distributed crystals of plate-like form grains with porous regions between them. It is clear from the figure that the grain boundaries are in touch as to form weak bonds between each other.

We observed a noticeable change of the grain size in SEM study. The difference in the grain size and the distribution of grains on the surface of the samples indicate the influence of different wt% of Li on the morphology of the samples. The SEM image of undoped specimen shows very small size grains with thin plake like structures, indicating poor formation of the superconducting phase (Fig. 7A). We also observed that the undoped sample has more voids. On the contrary the size of grains was larger in Li added samples, the difference is clearly visible from SEM images: the grain size is the smallest in sample A and the largest in sample E.

The theoretical density of BPSCCO system is about 6.3 g/cm<sup>3</sup> obtained from the lattice parameters [17]. The density of pellets in this work was determined to be 5.4881 g/cm<sup>3</sup>, 5.4529 g/cm<sup>3</sup>, 5.4377 g/cm<sup>3</sup>, 5.1934 g/cm<sup>3</sup>, and 5.1482 g/cm<sup>3</sup>, respectively, for  $x = 0.00$  to  $x = 0.20$ , measured by water displacement Archimedes' method. The bulk densities determined by the Archimedes technique is in the 82 to 87% range of theoretical density. This shows that about two-thirds of the pores were filled by water during the density determinations. This result is in accordance with the porous structure of ceramic superconductors.



**Fig. 6** Temperature dependence of the real part of AC susceptibility of the samples  $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_{3-x}\text{Li}_x\text{O}_y$  (A)  $x = 0.00$ , (B)  $x = 0.05$ , (C)  $x = 0.10$ , (D)  $x = 0.15$ , and (E)  $x = 0.20$



**Fig. 7** SEM images of the samples  $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_{3-x}\text{Li}_x\text{O}_y$  with: (A)  $x = 0.00$ , (B)  $x = 0.05$ , (C)  $x = 0.10$ , (D)  $x = 0.15$ , and (E)  $x = 0.20$

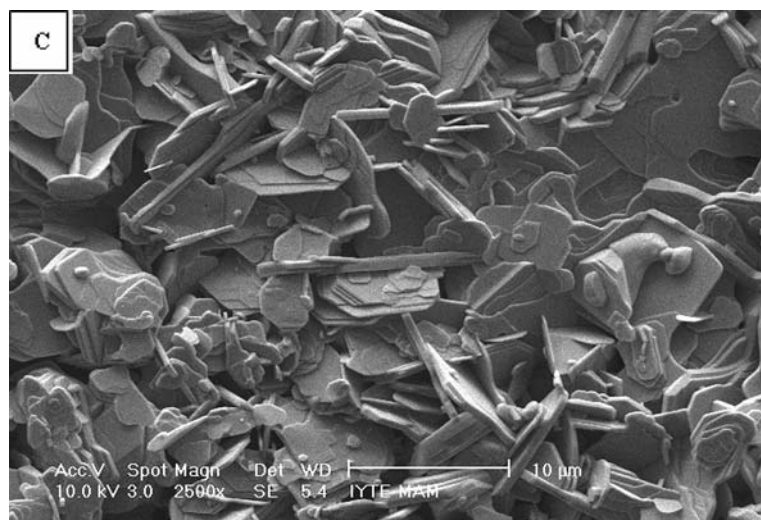
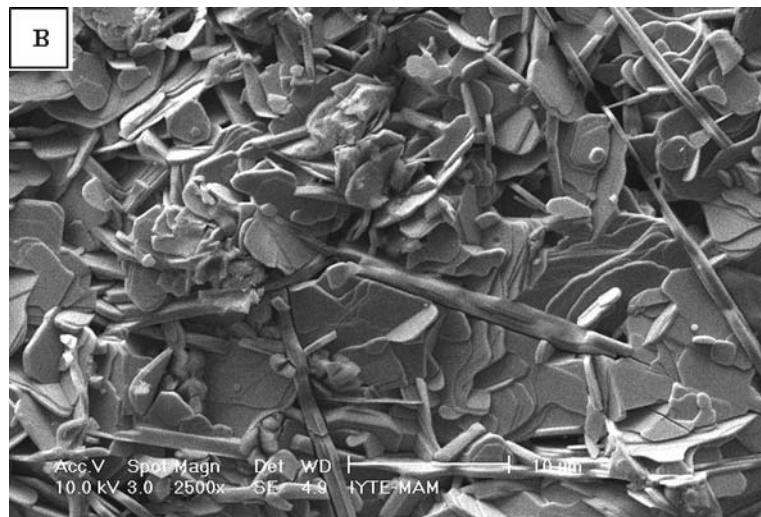
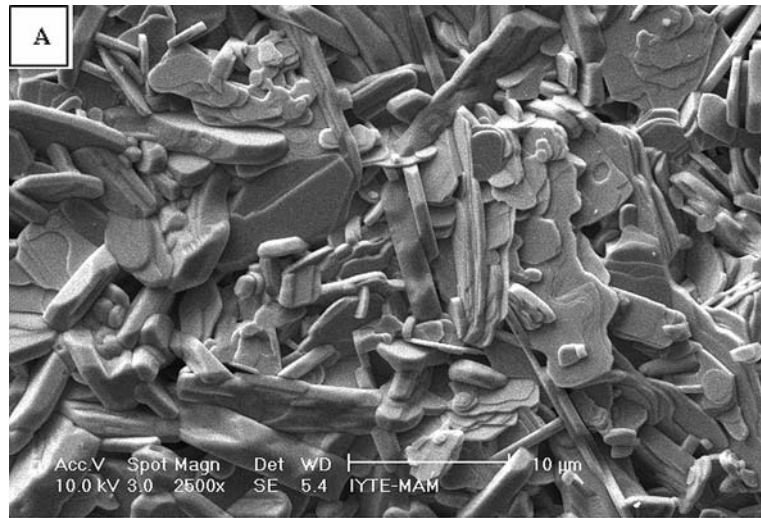
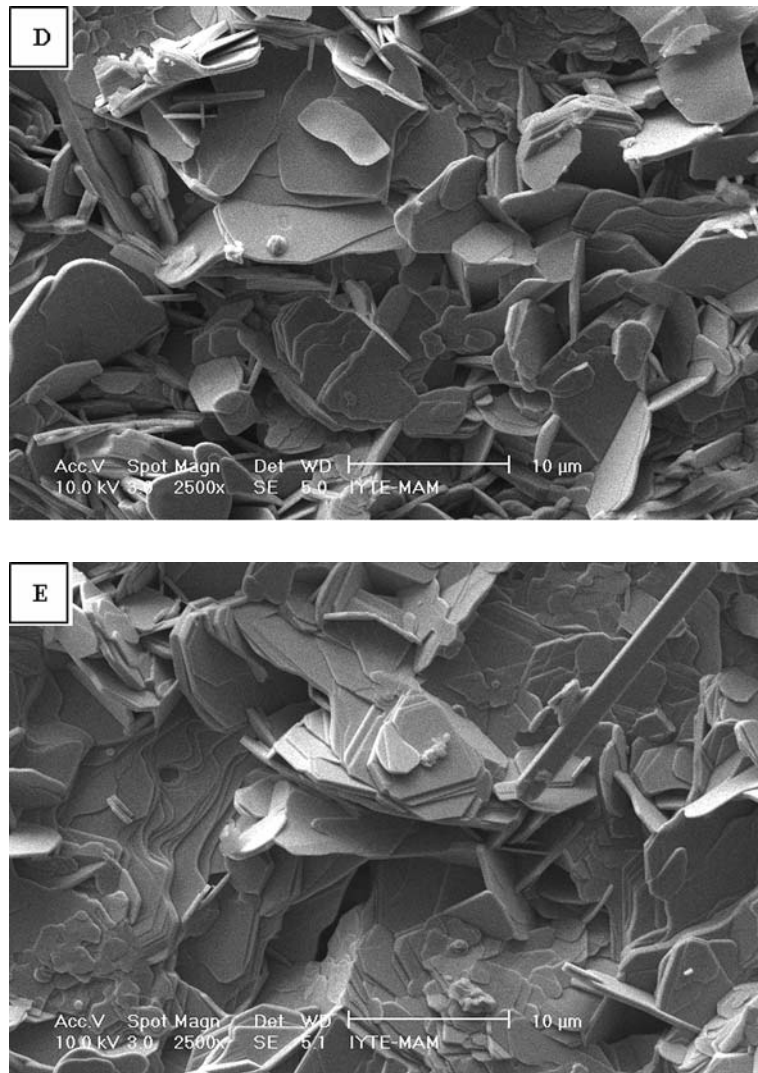


Fig. 7 (Continued)



#### 4 Conclusion

The samples with nominal composition  $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_{3-x}\text{Li}_x\text{O}_y$  were prepared by the solid-state reaction method. The systematic investigation of the influence of Li doping on the phase formation and superconducting state properties of (Bi,Pb)–Sr–Ca–Cu–O superconductors revealed that the high- $T_c$  (2223) phase in the (Bi,Pb)–Sr–Ca–Cu–O system by means of Li addition in the form  $\text{Li}_2\text{O}$  can be obtained. The room temperature X-ray diffraction indicated the formation of large amount of Bi-(2223) phase along with minor amount of Bi-(2212) phase. The volume fraction formed was estimated from the X-ray intensities of Bi-(2223) and Bi-(2212) phases. The analysis showed that the samples with  $x = 0.05$  and  $x = 0.20$  had the highest volume fraction of the Bi-(2223) high- $T_c$  phase and was 81%. The onset critical temperature  $T_c$  (onset) and zero electrical resistivity temperature  $T_c$  ( $R = 0$ ) were determined from the electrical resistivity measurements. The critical temperature

for  $x = 0.20$  at zero resistance exhibits a maximum critical temperature ( $T_c = 98$  K), whereas the transition width shows a minimum. The DC electrical data agree with AC magnetic susceptibility measurements.

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