

Structural characterization and superconductivity in $\text{Bi}_{1.7}\text{Pb}_{0.3-x}\text{Tb}_x\text{Sr}_2\text{Ca}_3\text{Cu}_4\text{O}_y$. The influence of Tb-doping

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$\text{Bi}_{1.7}\text{Pb}_{0.3-x}\text{Tb}_x\text{Sr}_2\text{Ca}_3\text{Cu}_4\text{O}_y$ compounds ($0.0 \leq x \leq 0.1$) are synthesized by using the conventional high temperature melt quenching technique to study the influence of Tb substitution on their high- T_c superconducting properties. The effects of different Tb doping levels on the superconductor structure have been investigated by electrical resistance, magneto-resistance, scanning electron micrographs, and XRD measurements. It has been observed that, with increasing Tb^{3+} substitution for Pb^{2+} the high- T_c Bi-(2223) phase gradually transforms into the low- T_c Bi-(2212) and Bi-(2201) phases. It should be pointed out that the low- T_c superconducting phases of Bi-(2212) and Bi-(2201) are formed for all the doping levels. The data suggest that with the increasing Tb^{3+} doping level the superconductivity in $\text{Bi}_{1.7}\text{Pb}_{0.3-x}\text{Tb}_x\text{Sr}_2\text{Ca}_3\text{Cu}_4\text{O}_y$ system is suppressed due to the destruction of the phase coherence by pair-breaking effects, such as magnetic. The magneto-resistance results clearly demonstrate that with increasing magnetic field first the high- T_c Bi-(2223) phase gradually diminishes and almost the low- T_c Bi-(2212) phase remains, however, the trends indicate that at a reasonably high magnetic field the low- T_c phase will also be destroyed and hence the superconductivity will not be observed any more, as expected.

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1. Introduction

An interesting research area of superconductivity receiving a great deal of attention is the substitutional studies. Several properties of high- T_c superconductors have been identified as being technologically critical. These properties are a high critical current density, J_c , a high transition temperature, T_c , good thermoelectric power (TEP) and mechanical properties. There have been several reports on microstructural, electrical and physical properties of Bi-(Pb)-Sr-Ca-Cu-O superconducting systems [1-10]. Many workers have observed that the substitution acts as a probe of the chemical and structural environment which determines whether or not the system exhibits superconductivity [11]. These studies explain the decrease in T_c for some doping materials with increasing substitutional concentration, and the subsequent metal-insulator cross over, due to the decrease in the average valance of the copper, brought about by the excess charges induced by rare earth ions, i.e., doped ions. In some systems a so called superconductor-insulator transition has been also observed [12-14] which is said to be caused by an enhanced insulating nature of the Bi-O planes and a decrease in O, $2p$ hole concentration in the Cu-O planes.

Since the superconductivity in the Bi-based systems is believed to be associated with the Cu-O₂ planes, most of the substitutional studies have been made at the Cu site [15, 16]. A consideration of the relative valences and radii suggests that the 3d transition metals seem to be the most attractive elements to substitute for Cu in these materials. All doping elements so far investigated lower the

transition temperature. The mechanisms proposed as being responsible for the decrease and eventual suppression of the transition temperature in the copper oxide based superconducting systems are magnetic pair breaking, a change in the oxygen vacancy positions and local symmetry, and a change in electronic mechanisms such as band filling and electronic localization [17-19].

A study on thin films of doped Y-123 has shown that partial substitution is possible for Tb and a depression of T_c has been observed from 90 K to 55 K on T_c up to the solubility limit of 50% in thin films [20] and 30% in bulk powders [21]. Considering Tb to be trivalent, the low solubility of Tb in Y-123 cannot be due to the ionic radius but is probably caused by the stability of other phases that contain tetravalent Tb [22].

The intercalation process is a well-known technique for improving the properties of the superconducting systems. Interesting results have been reported due to the doping or intercalation of Pb to BSCCO system, that enhances the high- T_c phase formation and mechanical characteristics [23-25]. In this manner, the properties of BSCCO can be governed by the doping or intercalation of the elements having a different ionic radii and a different bonding character. Rare earth elements could be the candidate for the intercalation, since their ionic radii include those of Pb, Bi, Sr and Ca [26]. In view of this, it is of interest to investigate the effect of substitution of Tb having 3+ valance state and with ionic radius 0,98 Å on the properties of BSCCO system. These substitutions will also enable the study of the dependence of T_c on the oxygen excess as well as the doping concentrations in detail. In this study, the preparation and properties of

$\text{Bi}_{1.7}\text{Pb}_{0.3-x}\text{Tb}_x\text{Sr}_2\text{Ca}_3\text{Cu}_4\text{O}_y$ systems are examined by substituting Tb^{3+} ions at the Pb^{2+} sites. The role of the dopant and its influence on the stability as well as on the superconducting properties of Bi-based superconductors are discussed in terms of XRD, SEM, resistivity and magneto-resistance characterizations.

2. Experimental procedure

For our experimental studies, Tb-added $\text{Bi}_{1.7}\text{Pb}_{0.3-x}\text{Tb}_x\text{Sr}_2\text{Ca}_3\text{Cu}_4\text{O}_y$ ($x=0.0, 0.025, 0.050, 0.075, 0.100$) superconductors were prepared by the traditional solid state synthesis. For the compounds appropriate amounts of the starting mixture of oxides and carbonates of the respective elements were calcined at $750\text{ }^\circ\text{C}$ for 12 hours. The resulting bulk materials were crushed and ground in acetyl-acetone. Then the materials were dried and again calcined at $750\text{ }^\circ\text{C}$ for another 12 hours. The well calcined samples were re-crushed and ground. The resulting powders were then pressed into pellets of 10 mm diameter by applying a force of 4 tons. Heat treatment was performed in two stages. At the first stage, all samples were sintered at $840\text{ }^\circ\text{C}$ for 170 hours in air and cooled down to the room temperature in the furnace. All the samples were then ground and again pelletized under the same force and sintered again at $840\text{ }^\circ\text{C}$ for 170 hours in air to achieve good superconductivity with higher T_c . Hereafter, the samples with x values of 0.000, 0.025, 0.050, 0.075 and 0.100 are labeled as A, B, C, D and E, respectively. X-ray powder diffraction analyses were performed by using Rigaku RadB powder diffractometer system with $\text{CuK}\alpha$ radiation and a constant scan rate between $2\theta=3^\circ\text{-}60^\circ$. The microstructural and compositional characterizations were carried out with LEO Evo-40 VPX scanning electron microscope (SEM). Resistivity measurements, under different magnetic fields, were carried out using Cryogenic Q-3398 Vibrating Sample Magnetometer (VSM) system.

3. Results and discussion

3.1. Electrical results

The resistivity (ρ) versus temperature (T) curves for the samples, from 300 K to down 15 K, are shown in Fig. 1. All the samples show a metallic behavior in their resistivity above their respective superconducting transition temperatures. For the sample A although the $T_{c.onset}$ of superconductivity is 89 K, resistivity goes to zero at $T_{c.offset} = 83$ K. Hence, there is a superconducting transition range ΔT of about 6 K and the (2212) phase is the dominant phase in the sample A, as it is explained in Sec. 3.3.

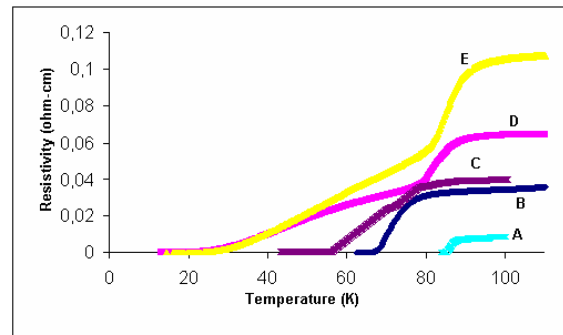


Fig. 1. The resistivity versus temperature curves of the samples A, B, C, D and E.

For the sample B, the normal state resistance value was found to be considerably increased relative to that of the sample A. Below T_c an unstable resistance behavior is observed. We believe that this unstable behavior may arise from a deformation in the short-range order due to the substitution of magnetic Tb^{3+} ions into the BSCCO system. For this sample, $T_{c.onset}$ and $T_{c.offset}$ values are found to be 82 K and 67 K, respectively. For the sample C, the $T_{c.onset}$ and $T_{c.offset}$ temperatures are found to be 80 K and 54 K, respectively.

Table 1. Structural and electrical properties of the $\text{Bi}_{1.7}\text{Pb}_{0.3-x}\text{Tb}_x\text{Sr}_2\text{Ca}_3\text{Cu}_4\text{O}_y$ samples.

Samp.	Temp. ($^\circ\text{C}$)	Time (h)	Symmetry	a(=b) (\AA)	c (\AA)	$T_{c.onset}$ ($^\circ\text{K}$)	$T_{c.offset}$ ($^\circ\text{K}$)	ΔT ($^\circ\text{K}$)	P, Hole number
A	840	170	Tetragonal	5.39	30.8	89	83	6	0.208
B	840	170	Tetragonal	5.39	30.8	82	67	15	0.192
C	840	170	Tetragonal	5.40	30.7	80	54	26	0.196
D	840	170	Tetragonal	5.40	30.7	87	27	60	0.180
E	840	170	Tetragonal	5.41	30.7	89	26	63	0.171

For the sample D again a metallic behavior down to T_c , was detected, however two phase transitions were

observed at lower temperatures. The transitions at 87 K and 79 K point out that the sample contains mixed phases

of Bi-(2212) and Bi-(2201). According to the XRD results given in Sec. 3.3., the dominant phase in this sample is Bi-2201. The $T_{c,offset}$ value was found to be 27 K. When the Tb amount was increased to $x=0.1$, sample E, the material was found to be still metallic in the normal state. For the sample E, two step transitions were obtained at 89 K and 80 K and the $T_{c,offset}$ value was found to be 26 K, after a long tail.

The values of $T_{c,onset}$ and $T_{c,offset}$ are given in Table 1. It is obvious that $T_{c,offset}$ values decrease with increasing Tb doping, however, $T_{c,onset}$ values seen to show no definite trend. As a result, suppressed electrical properties were obtained in the Tb-substituted BSCCO system. The intergrowth of impurity phases in the BSCCO grains, the weak coupling between the impurities and BSCCO grains, and structural distortions may be the cause of a decrease especially in the $T_{c,offset}$ values and an increase on the normal state resistance value.

3.2. Hole concentration

The hole concentration, p , per Cu atom in high- T_c superconductor materials can be calculated by using the relation [27].

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

where T_c^{\max} is taken as 90 K for the Bi-2212 and 110 K for the Bi-2223 system. Equation (1) has been successfully applied to the doped/substituted high- T_c systems previously by many research groups [28-32]. Recently calculations for the unsubstituted Bi-2223 system have shown that the p -value ranges from 0.116 to 0.160. In this study, the p -values of the samples A, B, C, D and E have been calculated to be 0.208, 0.192, 0.196, 0.180 and 0.171 respectively and are given in Table 1. The results obtained show that the p -values of our samples seem to decreasing slightly with increasing Tb concentration, however, are not much higher than the unsubstituted Bi-2223 system, which points out that the amount of Tb dopant is far from the overdoping limit, in agreement with the results of Satyavathi et.al [33]. Therefore, the increase on the normal state resistance value of the samples and a decrease on the superconducting transition temperature, $T_{c,offset}$, can be attributed to hole filling mechanism, which was previously described by other groups [4,8].

3.3. X-Ray Diffraction results

The XRD patterns of the A, B, C, D and E samples are shown in Fig. 2. The sample A has both the high- T_c - (2223) phase and the low- T_c - (2212) phase, it also has non-superconducting phases (Ca-Sr-Cu-O and Ca-Pb-O). For the sample A, the low- T_c phase (2212) is the dominant one. Although the highest attainable transition temperature for the BSCCO system is 110 K, because of the existence of large amount of low T_c phases in our non-doped sample (sample A) we observed the superconducting transition temperature to be 89 K. The X-ray diffraction patterns for the samples B, C, D and E, indicates that increasing the amount of Tb^{3+} ions in the Tb doped samples slightly worsens the superconducting phase and the

superconducting properties. The main phases of the samples B, C, D and E appear to be (2212) and (2201) phases. As a result of increase in Tb content in the samples the high T_c phase has disappeared. When the amount of the Tb^{3+} ions in the sample is greater than the 0.01, the structure of the samples seems to show some changes as can be deduced from the X-ray intensities of the phases. As a result, the XRD studies have revealed that with the increasing Tb^{3+} ions in the samples, high- T_c - (2223) phase has gradually transformed into the (2212) phase, but also some non-superconducting and impurity phases have also appeared. Hence, it can be concluded that the volume fraction of the high- T_c - (2223) phase decreases with increasing amount of Tb.

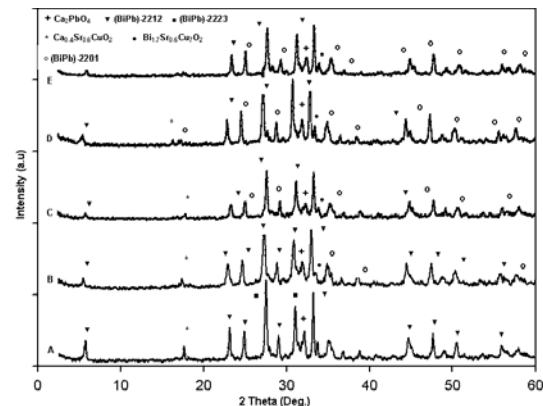


Fig. 2. XRD patterns of the the $Bi_{1.7}Pb_{0.3-x}Tb_xSr_2Ca_3Cu_4O_y$ samples. A) $x = 0.0$, B) $x = 0.010$, C) $x=0.050$, D) $x=0.075$ and E) $x=0.100$

3.4. Structural analysis

It is obvious that when Tb is substituted into the Bi- (2223) system, the phase coordination is destroyed and multiphase, complex and deformed structures are formed. The crystal symmetry of all the samples is found to be tetragonal. The calculated unit cell parameters of the samples are presented in Table-1. and the variation of them with the substitution, x , is shown in Fig. 3. As seen in Table-1. and shown in Fig. 3, with increasing x , a monotonic decrease of the c - parameter with simultaneous elongation of the a - parameter have been observed and this correlates with the increasing hole concentration in the CuO_2 plane, as suggested by Satyavathi et. al and Zandbergen et. al. [33,34]. It is believed that Tb^{3+} goes into the bismuth oxide layers in the main matrix and increases the oxygen content. This produces a distortion on the lattice structure; while increasing the a -axis at the same time decreases the c - axis parameter. A slight increase in cell volume was observed, in contrast to the Sm substituted samples where it remained almost the same up to $x = 0.100$ and increased thereafter [35]. One can conclude that with the addition of Tb, the hole concentration increases leading to an increase in the normal state resistivity, as expected. Similar results were previously obtained by other research groups for BSCCO material [36-38]. We have found no impurity phase other

than CuO , which possibly came through the copper plates during quenching process and not dissolved completely in the matrix during the heat treatment cycles.

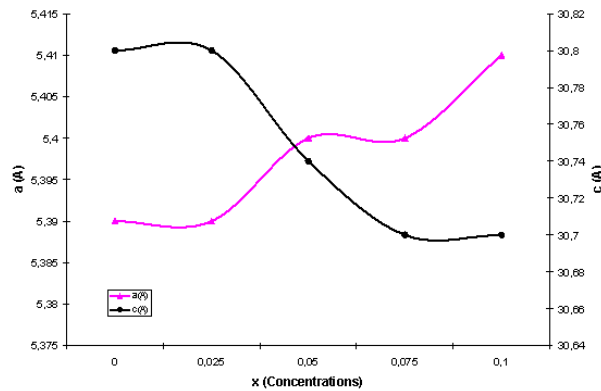


Fig. 3. The variation of the unit cell parameters with x concentrations.

3.5. SEM results

The scanning electron microscope photographs for the samples A,B,C,D and E are shown in Fig. 4. SEM image of the undoped sample (sample A) indicates that crystallization process is not completed. The sample contains some impurities which are not diffused into the structure. This is most probably either due to the thermal process duration or to the improper setting of the sintering temperature to obtain good crystallized superconducting sample. This conclusion is confirmed with the obtained low T_c values. Furthermore, it is observed that doping with Tb^{3+} ions causes a change in the structure and as a result forces the samples to have lower T_c phases. With increasing amount of Tb^{3+} ions, a decrease takes place in the grain sizes, and whereas, an increase is observed in the number of porosities. When Tb doping ratio is greater than 0.010 some new impurity phases simultaneously form. All these observations lead us to conclude that the increasing Tb^{3+} doping concentration negatively affects the superconducting properties of the samples, studied.

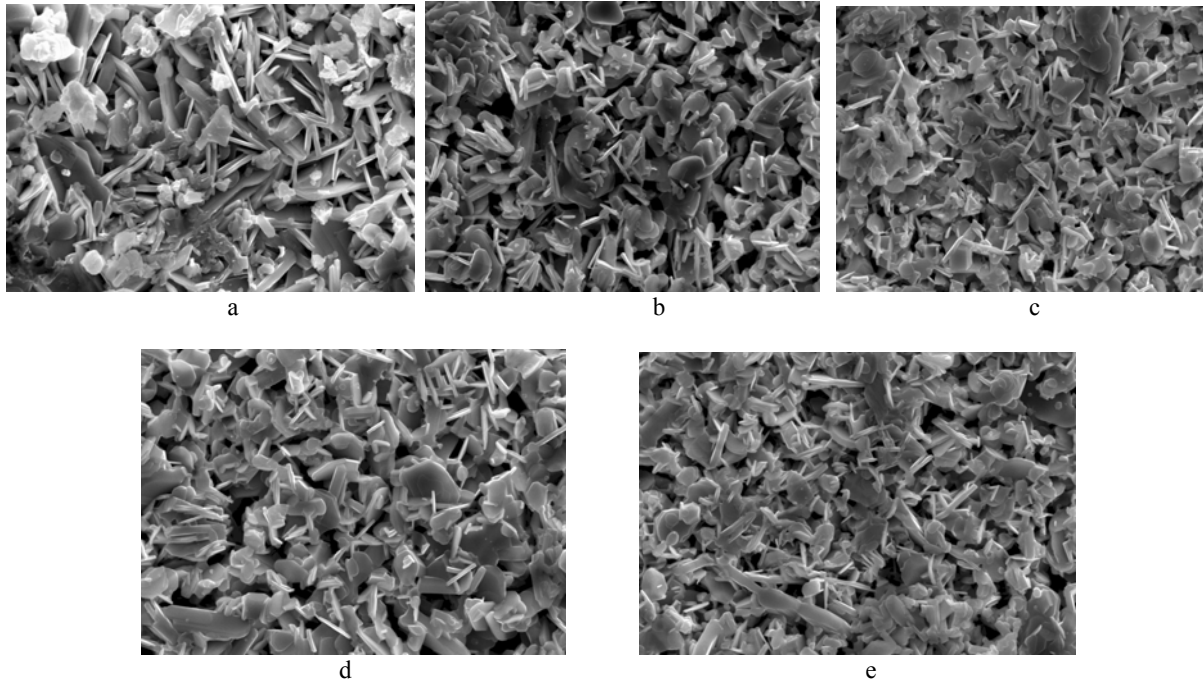


Fig. 4. SEM photographs of the samples A, B, C, D and E, respectively. (20 kV, X2000, $10\mu\text{m}$)

3.6. The magnetic field dependence of resistivity

Figs. 5A-E depict the temperature dependence of resistivity, ρ , measurements in magnetic fields between 0 and 6 Tesla. Zero field cooling procedure was used for all the measurements. It has been obtained that all the resistivity values measured at zero magnetic field, decrease almost linearly with decreasing temperature in the normal state, and at lower temperatures show two superconducting onset transition temperatures. This

behavior points out that the samples have multi phases, namely Bi-(2223), Bi-(2212) and/or Bi-(2201). The Bi-(2223) phase smears out with increasing magnetic field and hence almost the Bi-(2212) and Bi-(2201) phases remain for all the samples at 6 T, indicating that at a reasonably high magnetic field only the Bi-(2212) and Bi-(2201) phases will remain. Table-2. shows values of $T_{c.onset}$ and $T_{c.offset}$ under different applied magnetic fields. As it can be seen from the Table-2, while the $T_{c.onset}$'s remain almost unchanged $T_{c.offset}$'s decrease with

increasing applied magnetic field. These temperatures are comparable with the irreversibility or depinning temperatures [28]. It is well known that in type-II superconductors, electric field arises, electrical resistance and hence energy dissipation occurs when the driving Lorentz force per unit volume, $F_L = \mathbf{J} \times \mathbf{B}$, exceeds the

pinning force per unit volume F_p . At lower temperatures, a higher magnetic field is needed to depin the flux line since, lowering the temperature increases the pinning force. This results in a shift of the zero resistance temperature or the irreversibility temperature [33,39,40] downwards as the magnetic field increases.

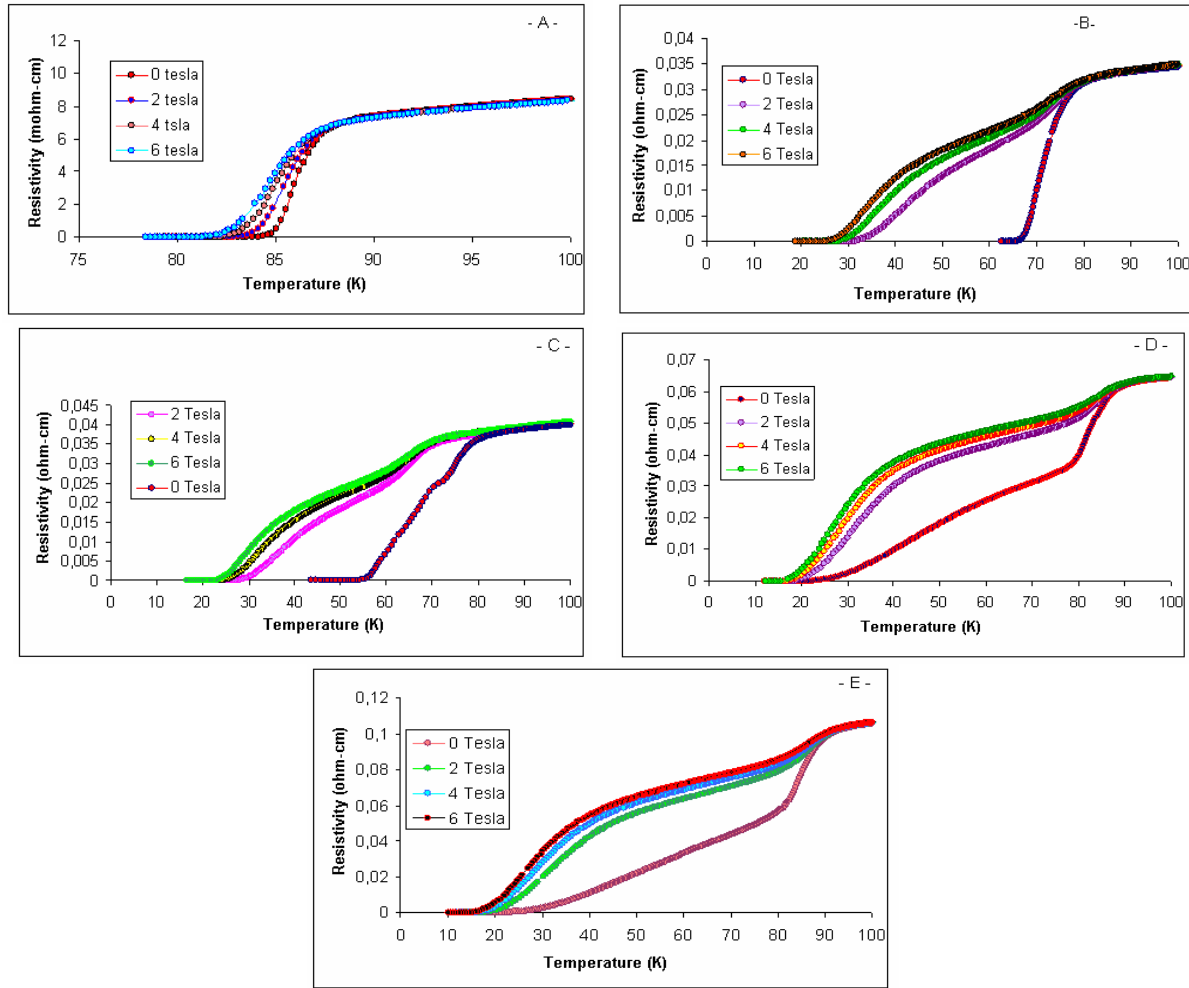


Fig. 5A-E. The influence of applied magnetic fields on the Resistivity versus Temperature plots for the samples A, B, C, D and E.

Table 2. The magnetic field dependence of resistivity.

Conc. x	0 Tesla		2 Tesla		4 Tesla		6 Tesla	
	$T_{c.onset}$ (° K)	$T_{c.offset}$ (° K)	$T_{c.onset}$ (° K)	$T_{c.offset}$ (° K)	$T_{c.onset}$ (° K)	$T_{c.offset}$ (° K)	$T_{c.onset}$ (° K)	$T_{c.offset}$ (° K)
0.00	89	83	87	82	87	81	86	81
0.025	82	67	80	31	78	27	77	25
0.050	80	54	70	28	68	25	67	24
0.075	87	27	87	21	86	18	86	17
0.100	89	26	89	20	88	17	85	16

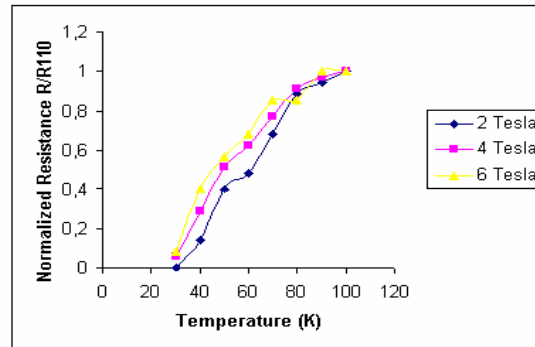


Fig. 6. Normalized resistance (R/R_{110}) against temperature for the sample B in external magnetic fields of 2, 4 and 6 Tesla.

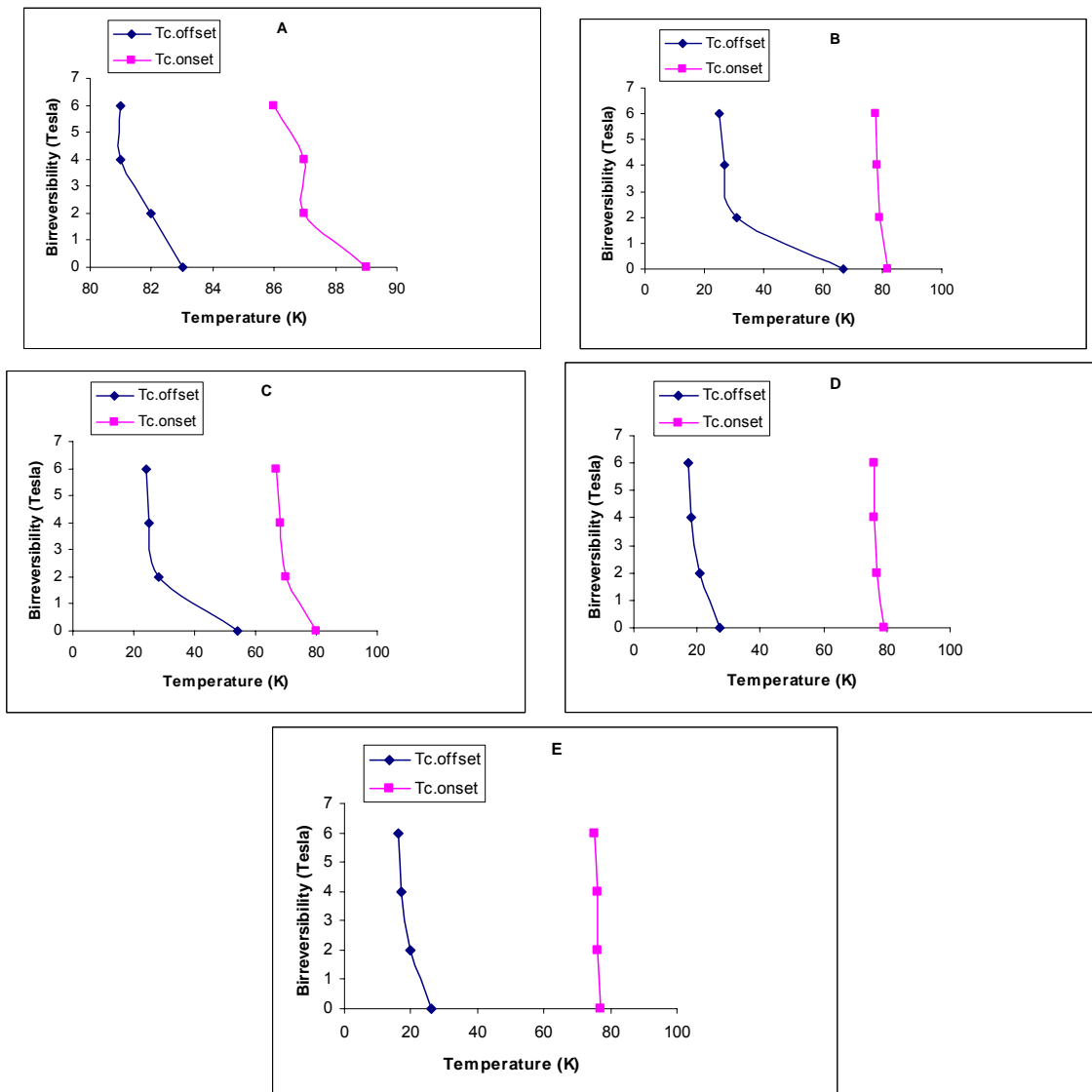


Fig. 7A-E. A comparison and variation of the irreversibility magnetic fields against the $T_{c,onsets}$ and $T_{c,offsets}$ of the samples.

3.7. The normalised resistance and the irreversibility

The graph of the normalized resistance R/R_{110} versus temperature T , obtained between the onset and offset temperatures have been displayed in Fig. 6, for the sample B, in order to see the effect of applied magnetic field B_a on resistance. As can be seen from the figure 6, the resistances shift towards lower temperature values with increasing field, indicating that the superconductivity is destroyed with increasing magnetic field, as expected. Figures 7A-E show the irreversibility magnetic field values versus the onset and offset temperatures, for the samples A, B, C, D and E, deduced from the magneto-resistance data given in Figs. 5A-E. It is obvious that while the variation of onset temperature is about 5 K, the variation of offset temperature is about 42 K within the studied magnetic field range. All these results strongly suggest that with a high enough magnetic field the superconductivity can be completely diminished, as expected. It is also known that the superconductivity is destroyed by increasing the amount of substituting materials like Nd, Gd, Sm etc., [5,6,7] in BSCCO superconducting materials. The formation of weak coupling between the impurity and superconducting grains and the intergrowth of the impurity solid solution phases also play a crucial role in diminishing the superconductivity. However, an optimum applied magnetic field can hinder the negative effects arising from the impurities on the superconductivity. Since the negative effect arising from the magnetic field is more than that of the impurities. The considerable decrease of the offset temperatures with increasing magnetic field supports this view (see figs 7A-E). Thus, it can be concluded that the effects of the impurities are obscured by the magnetic field.

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