

Transport and magnetic properties of Bi(Pb)2212 superconducting ceramics doped by low rate of potassium

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Samples of $\text{Bi}_{1.5}\text{Pb}_{0.5}(\text{Sr}_{1.8-x}\text{K}_x)\text{CaCu}_2\text{O}_{8+d}$ and $\text{Bi}_{1.5}\text{Pb}_{0.5}\text{Sr}_{1.8}\text{CaCu}_2\text{O}_{8+d}\text{K}_x$ have been prepared from powders of carbonates and primary oxides using the solid state reaction method and compared in this study. In the first case, potassium (K) is substituted in the strontium (Sr) site. In the second one, K is added. A part of the paper is devoted to discuss the results obtained by X-ray diffraction (XRD) analysis and scanning electron microscope (SEM) observations of $(\text{Bi,Pb})_2\text{Sr}_{1.8}\text{CaCu}_2\text{O}_{8+d}$ samples doped by potassium to provide additional microstructural information related to the doping method. These analyses are supplemented by resistivity and magnetic measurements. Results show how the rate of doping by potassium affects transport and magnetic properties of Bi(Pb)2212 phase. The critical current density (J_c) is improved using the two methods and it reaches a double value by the addition of K as compared to the undoped sample.

Keywords: *ceramics; superconductors; transport properties; magnetic properties*

1. Introduction

One of the most fascinating manifestations of quantum mechanics is superconductivity. It is used in many fields: scientific research, nuclear fusion, nuclear magnetic resonance medical imaging, storage of electrical energy and in transport (trains with magnetic levitation but also magneto-hydrodynamic propulsion). It is characterized by two remarkable properties: zero resistance and expulsion of magnetic field (Meissner effect). The discovery of high critical temperature (HTSC) superconductors by J.G. Bednorz and K.A. Müller in 1986 [1], allowed the transition of technologies based on low critical temperature superconductors to HTSC superconductors and extend the applications of superconducting materials. Bismuth-based compounds have the general formula $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_{4+2n+d}$ with $1 \leq n \leq 3$ (Bi2201-Bi2212-Bi2223) [2–5]. The role

of bismuth substitution by lead in the phase Bi2212 is to increase the T_c and the critical current density (J_c). Lead (Pb) changes density holes in the charge reservoir constituted by the BiO layer. The effect of lead can thus serve as moderator or amplifier when a second substitution is performed either at CuO_2 planes or at other planes (Sr or Ca). The role of lead appears also in suppressing the superstructure [6]. Chemical doping in high T_c superconducting cuprates (HTSC) have been reported at many studies [7, 8]. The primary purpose of the authors was to improve superconducting properties of their compounds [9–12]. The rare-earth element in many HTSC families of compounds plays an important role for the stabilization of the structure [7, 13, 14]. For more significant results, small concentrations of doping atoms should be considered. After the discovery of the Bi-2212 ($\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+d}$) superconducting phase [3, 5], the effect of doping by alkaline elements was studied [15–17]. This kind of doping results in changing the unit crystallographic cell parameters

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accompanied by a lowering of the phase formation temperature caused by the flux action of the alkaline element, for example: doping by potassium on the Sr site results in a contraction of the c axis parameter [16]. The decrease of oxygen content explains the generally observed enhancement of T_c [18]. More recently, in polycrystalline bulks of $\text{Bi}_2\text{Sr}_2\text{Ca}_{1-x}\text{K}_x\text{Cu}_2\text{O}_{8+d}$, ac susceptibility measurement revealed the optimization of intergrain connections. An improvement of critical current density has been noticed [19]. Addition of potassium allowed also a faster growth rate of Bi-2212 whiskers [20]. In this paper, we study the effect on the Bi(Pb)-2212 phase at low rate of potassium doping by substitution or addition.

2. Experimental

Samples of $\text{Bi}_{1.5}\text{Pb}_{0.5}(\text{Sr}_{1.8-x}\text{K}_x)\text{CaCu}_2\text{O}_{8+d}$ ($0 \leq x \leq 0.05$) were prepared with the usual method of solid state reaction using high grade purity powders of Bi_2O_3 , SrCO_3 , CaCO_3 , PbO , CuO and KOH . The starting mixtures were calcined at 800°C for 30 h. After that, the obtained powders were ground and pressed into pellet shape (13 mm in diameter and 1 mm thickness) under pressure of 5 ton/cm^2 and finally sintered at 850°C for 40 h. Preparation of samples of $\text{Bi}_{1.5}\text{Pb}_{0.5}\text{Sr}_{1.8}\text{CaCu}_2\text{O}_{8+d}\text{K}_x$ ($0 \leq x \leq 0.05$; addition method) follows the same route as in substitution but without KOH , which is added only after calcination. x : corresponds to the weight fraction of the powder of the free K Bi(Pb)-2212 phase obtained after calcination. Obtained samples were characterized by X-ray diffraction (XRD) using $\text{CuK}\alpha$ radiation and scanning electron microscopy (SEM) [21]. Indexation of XRD patterns and determination of lattice parameters of samples have been done using CELREF software. Resistivity of samples was measured in temperature range 10 K to 125 K, on a closed cycle helium refrigerator (Cryodine CTI-Cryogenics) using a standard DC four-probe technique with a constant current of 5 mA at Physics Department of Abant İzzet Baysal University of Bolu in Turkey. The magnetic measurements have been performed by means of a 9 Tesla Quantum Design PPMS (Physical

Properties Measurement System) equipped with a Vibrating Sample Magnetometer (VSM) option at Physics Department of University of Salerno in Italy. It is worth to underline that, before each measurement, the residual magnetic field entrapped in the superconducting magnet was reduced below $1 \times 10^{-4}\text{ T}$ [22, 23]. The $M(T)$ and $M(H)$ measurements of magnetization as a function of the temperature and the magnetic field, respectively, were performed using the typical procedures [24–26]. In particular, the $M(T)$ measurements were performed in zero field cooling (ZFC) and field cooling (FC) conditions. More precisely, the sample was first cooled down to 40 K in the absence of field, then a magnetic field of 0.002 Tesla was switched on and the data were acquired for increasing temperature (ZFC) reaching the room temperature 120 K. After that, the sample was cooled down again and FC magnetization was acquired in the presence of field. For $M(H)$ measurements, the sample was first thermally stabilized to 4.2 K in the absence of magnetic field. Then, the field was ramped to reach +9 T, then back to -9 T , and finally to +9 T again, in order to acquire the complete $M(H)$ loop.

3. Results and discussion

In our previous work, both substitution and addition affect the intensity of main peak's (Bi,Pb)-2212 [21]. Traces of parasitic phases Bi-2201 and /or Ca_2PbO_4 are present in all the samples. In SEM micrographs, the typical lamellar structure of HCTS is present. We can notice that the addition of K gives a faster growth rate of Bi-2212 whiskers [21]. The ionic radius of K^+ ion is 1.38 \AA when its coordination number is 6 and reaches 1.51 \AA for a coordination number of 8 [27]. The Sr^{2+} one is 1.18 \AA and 1.26 \AA respectively for the same coordination numbers. Thus, c axis may increase when K substitutes on Sr site. The observed contraction of c axis may be due to a transfer of charges between the CuO_2 planes resulting from the difference of valence between K^+ and Sr^{2+} ions. When K is added to the Bi(Pb)-2212 phase c axis increases for most of the samples suggesting that K substitutes on the Ca site where the ionic radius of Ca^{2+} is 1.12 \AA (coordination number of

8). The same kind of charge transfer is present but giving a different result when the site changes. The difference of behaviour between addition and substitution also appears in the variation of the orthorhombic strain $(b-a)/(a+b)$ versus the content x of K shown in Table 1 and Table 2 where the variations of c axis are reported. The effect of K on the orthorhombic strain is higher for substitution where its increase corresponds to a decrease of parameter c . This means that substitution by potassium on outer plane (the Sr site) promotes the displacement of oxygen ions toward the CuO_2 planes. The excess of charge in these planes increases the Jahn Teller distortion effect on the oxygen octahedron surrounding the Cu^{2+} ion. This excess of charge also causes the contraction of the c axis. With the addition of potassium, the behaviour of the orthorhombic strain is contrary, and a possible substitution on Ca site instead of Sr site is considered.

A standard four-probe technique measures the electrical resistivity. Fig. 1 shows its variation as a function of temperature and doping. The results show the typical rapid resistive transition of superconductors where T_c is determined. In Fig. 1(b), the values of resistivity of sample with $x = 0.01$ are divided by 20 in order to get all the curves together.

Table 3 and Table 4 present the critical temperatures T_c^{onset} (beginning of the transition from the normal state to the superconducting one) and T_c^{off} (end of the state corresponding to $R = 0$) of the samples. T_c^{onset} for all the samples obtained by addition or substitution is lower than the K free sample. Overall oxygen content and the disorder in CuO_2 planes [28, 29] are mechanisms responsible for variation of T_c^{onset} . For substitution, T_c^{onset} is maximum at $x = 0.01$ and corresponds to a minimum of orthorhombic strain and a high value of c axis. Substitution by potassium at the Sr site leads to a decrease of T_c^{onset} . Similar results were found by T.H. Duc et al. [12], the authors found that both transition temperatures T_c^{onset} and T_c^{off} were decreased with bulk of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_{2-x}\text{K}_x\text{Ca}_2\text{Cu}_3\text{O}_{10+d}$ where $x = 0.00, 0.04, 0.08$ and 0.1); only in this study, two superconducting phases of Bi-2223 and Bi-2212

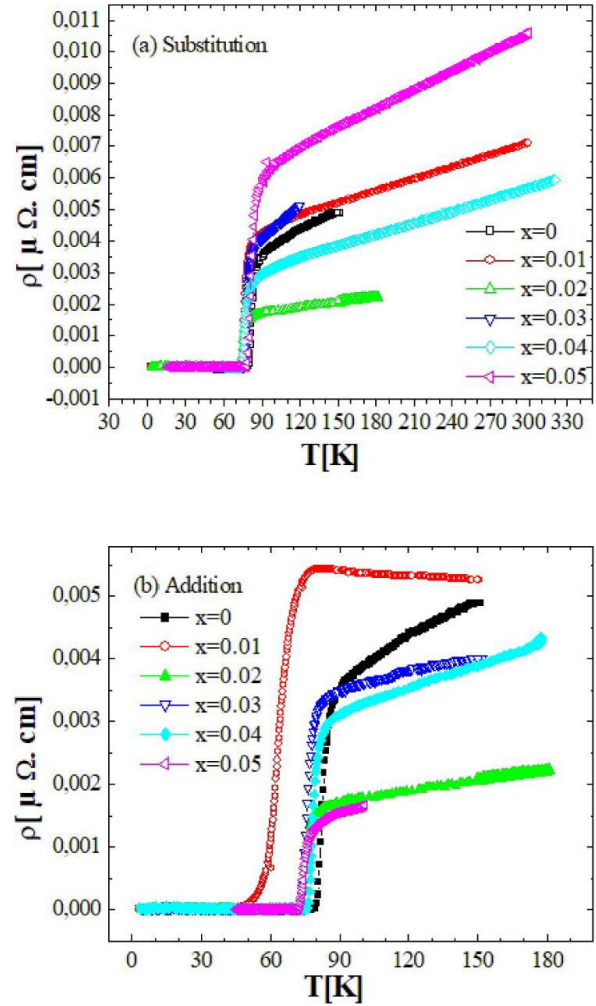


Fig. 1. Resistivity versus temperature curves obtained for samples doped by potassium substitution (a) and addition (b).

were formed. When a second substitution is used other than lead (Pb), T_c seems to behave otherwise. In case of superconducting K-Na co-doped $\text{Bi}_2\text{Sr}_2\text{K}_x\text{Ca}_1\text{Cu}_{1.75}\text{Na}_{0.25}\text{O}_y$ ($x = 0, 0.05, 0.1$ and 0.25) ceramics [17], the results show that T_c^{onset} values are gradually increasing as K content is increased. Our results are explained by the excess of charge in CuO_2 planes due to the oxygen octahedron surrounding the Cu^{+2} ion (Jahn Teller distortion effect); this causes a contraction of the c axis thus a decrease of T_c .

The used x rate of substitution of K is very low, its effect on the Bi(Pb)-2212 phase is not the same as on the Bi-2212 phase where higher rates were

Table 1. Variations of the orthorhombic strain $(b-a)/(a+b)$ versus x content of potassium for samples obtained by substitution and addition [21].

x	0	0.01	0.02	0.03	0.04	0.05
$(b-a)/(a+b) \times 10^3$ substitution	0.00413	0.00374	0.00552	0.00501	0.00375	0.00452
$(b-a)/(a+b) \times 10^3$ addition	0.00413	0.00412	0.00361	0.00429	0.00269	0.00439

Table 2. Variations of lattice parameter c versus x content of potassium for samples obtained by substitution and addition [21].

x	0	0.01	0.02	0.03	0.04	0.05
$c[\text{\AA}]$ substitution	30.6841	30.71	30.5705	30.5124	30.6947	30.7269
$c[\text{\AA}]$ addition	30.6841	30.7985	30.7479	30.7203	30.6479	30.7074

used [16]. A decrease of the T_c is observed contrary to the increase observed in the Bi-2212 phase. Table 4 gives values of T_c^{off} for all the samples. The results show that T_c^{off} for samples obtained by addition, is lower than the pure sample with a minimum at $x = 0.01$.

For those obtained by substitution, the same behaviour is observed and the T_c^{off} value corresponding to $x = 0.01$ is improved. T_c^{off} indicates the beginning of the flux creep of the vortices and gives information about the quality of the pinning in the sample. The critical temperature T_c of the samples is also determined from the $M(T)$ measurements in zero field cooling (ZFC) and field cooling (FC) conditions with an applied field of 0.002 T (see Fig. 2(a)–(b)). In this case, T_c is equal to the temperature corresponding to the onset of the ZFC magnetization drop as shown in Fig. 2(a)–(b). From the enlargement of the curve in the region of the superconducting transition, we can notice that T_c reported values are consistent with those obtained by measurement of resistivity versus temperature. The difference ΔM , between 60 K and 90 K of the ZFC and FC branches (see Fig. 3(a)–(b)), gives the irreversibility temperature T_{irr} of the samples. T_{irr} corresponds to the value of temperature where ΔM becomes equal to zero corresponding to the ZFC and FC overlap [30].

Fig. 4 reports T_c and T_{irr} values for the K doped samples prepared by substitution (Fig. 4(a)) and by addition (Fig. 4(b)). T_{irr} values are lower than T_c while the difference between the two parameters for the same concentrations is not significant and

seems to be the same except for $x = 0.01$, which appears more significant. Addition of K causes a decrease in both T_c and T_{irr} , more importantly in the $x = 0.01$ sample. On the other hand, substitution by K causes a quasi-linear decrease of T_{irr} until $x = 0.04$. At $x = 0.05$, the decrease is significantly lower giving a value near to that of the undoped sample. T_c is improved for $x = 0.01$ but it decreases for the other values of x , except for $x = 0.01$, T_c has a similar behaviour as T_c^{onset} obtained by transport measurement.

Fig. 5 shows, for the K doped samples by substitution (Fig. 5(a)) and addition (Fig. 5(b)), the magnetic hysteresis loops measured at $T = 4.2$ K with the applied field H parallel to the c axis ($H||c$). Use of the Bean critical state model [31, 32] allows to extract the critical current density J_c from the $M(H)$ curves. The formula used is:

$$J_c = 20\Delta MH/[b(1-b/3a)]$$

$$\Delta MH = Mdn - Mup$$

Where Mdn is the magnetization measured for decreasing, Mup for increasing, the applied magnetic field. a and b are the lengths (in cm) characterizing the cross section of the sample perpendicular to the applied field ($H||c$).

Fig. 6 reports J_c variations versus the applied field H for the K doped samples by substitution (a) and addition (b). Except for $x = 0.01$ doped by addition, both methods enhance J_c . Highest J_c is obtained in sample doped by substitution

Table 3. T_c^{onset} of the samples.

x	0	0.01	0.02	0.03	0.04	0.05
T_c^{onset} substitution [K]	82.19	81.94	79.03	79.14	75.28	80.4
T_c^{onset} addition [K]	82.19	70.85	78.3	79.38	80.31	78.72

 Table 4. T_c^{off} of the samples.

x	0	0.01	0.02	0.03	0.04	0.05
T_c^{off} substitution [K]	79.27	73.73	74.56	75.71	74.43	77.68
T_c^{off} addition [K]	79.27	47.31	73.78	73.56	76.13	72.05

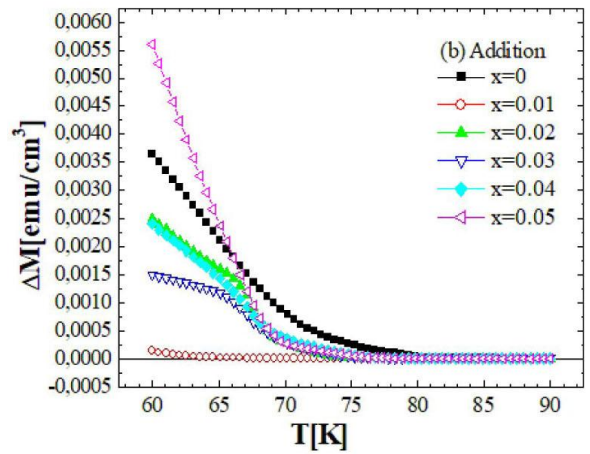
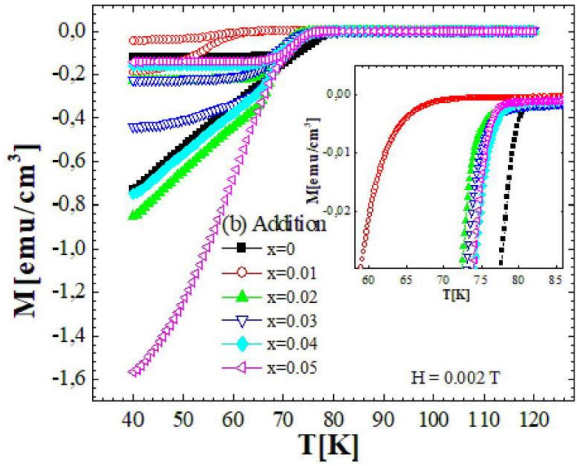
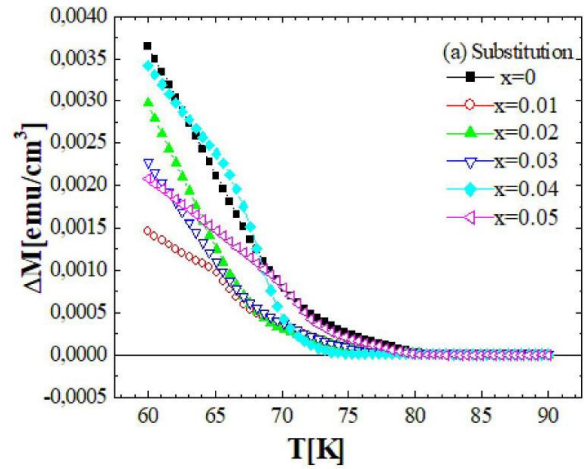
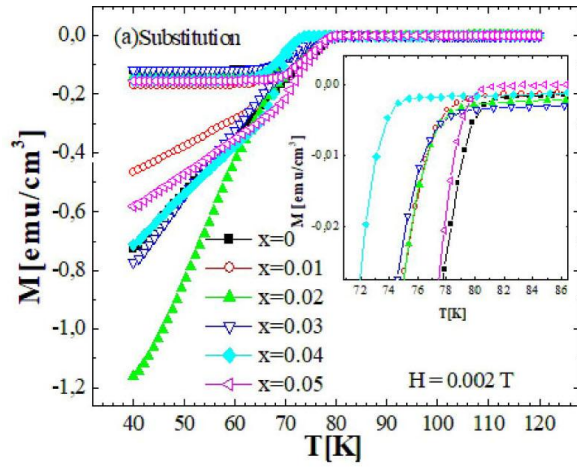

 Fig. 2. Magnetization as the function of temperature $M(T)$ using ZFC-FC procedure for the K doped samples by substitution (a) and by addition (b). The applied field is 0.002 Tesla. The insets show a magnification of the region near the superconducting transition.

 Fig. 3. Variation of ΔM with temperature for samples obtained by substitution (a) and addition (b) of potassium

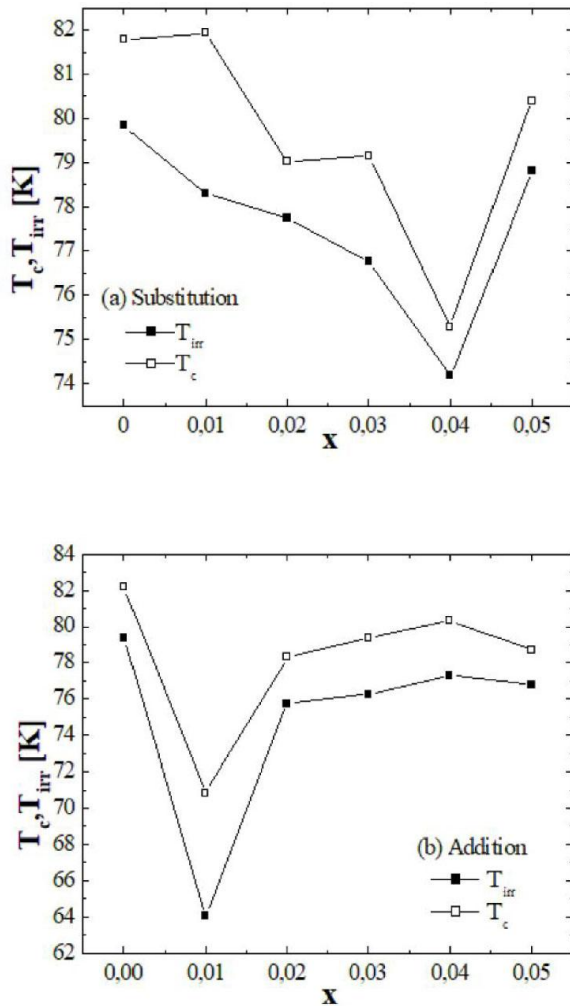


Fig. 4. T_c and T_{irr} values obtained for K doped samples by substitution (a) and addition (b) of potassium

with $x = 0.04$ and sample doped by addition with $x = 0.02$. Addition of K gives higher values of J_c reaching a maximum more than two times the undoped one. In Bi based superconducting ceramics, the structural properties are beside the anisotropy of physical properties the main factor governing the behaviour of J_c . Improvement of critical current density is due to the enhancement of flux pinning properties [33]. This depends at its turn on structure for intrinsic pinning and grain boundaries for pinning centres (low angle grain boundaries can act as pinning centres). Effective pinning centres are created by doping impurities, of the order of coherence length, in the system. This will lead to an improvement of flux pinning properties of

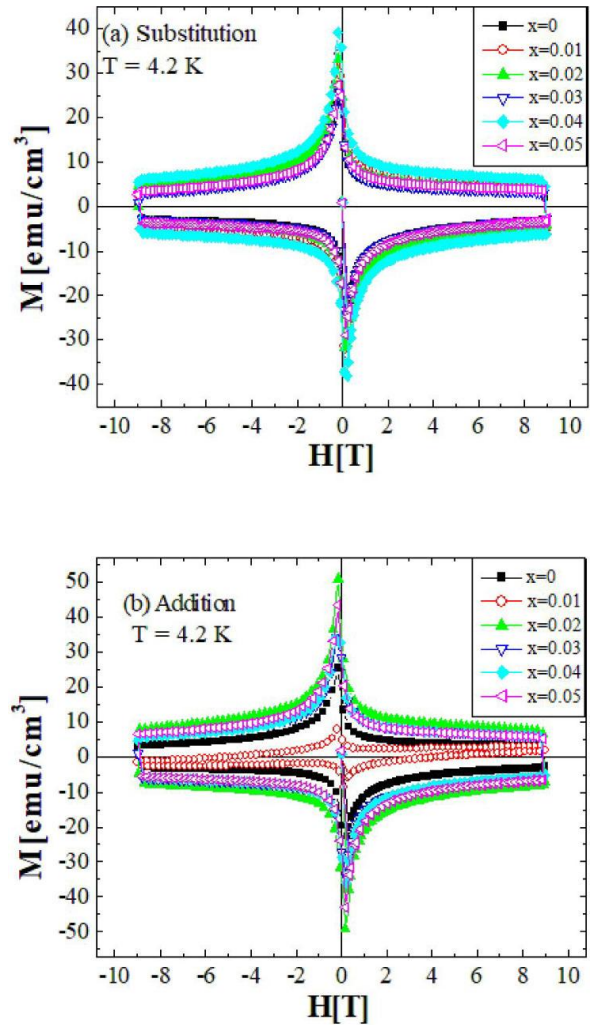


Fig. 5. Magnetic hysteresis loops measured at $T = 4.2$ K for the K doped samples by substitution (a) and addition (b).

the Bi system. Impurities chosen for flux pinning should not deteriorate the superconducting properties [34, 35]. J_c depends also on grain size and orientation, which control the size of superconducting current loops [19, 36]. The lowest value of J_c is obtained in the sample doped by addition with $x = 0.01$.

4. Conclusion

In the present work, samples of Bi(Pb)2212 are doped by potassium using two methods: substitution and addition. XRD and SEM analysis shows

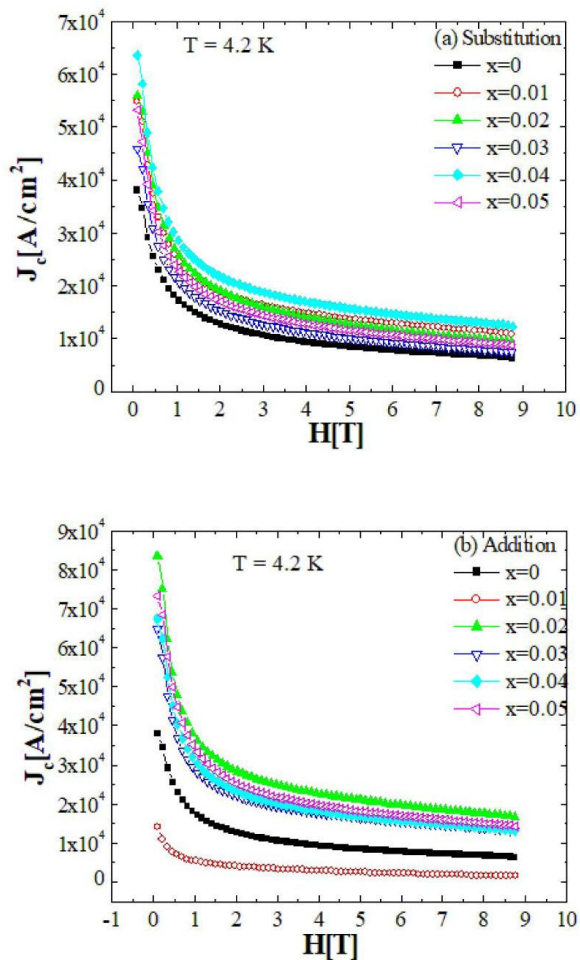


Fig. 6. Magnetic field dependence, at $T = 4.2$ K, of critical current density J_c for K doped samples with substitution (a) and addition (b).

that substitution by K enhances the texture, while for addition, this effect is obtained only for a lower rate of K [21]. Resistivity measurements show a decrease of T_c^{onset} and T_c^{off} for both methods. The K doping by substitution may increase the incorporation of oxygen in the structure; this will reduce the Cu valence and consequently a degradation of T_c^{onset} .

Magnetic measurements show an improvement of the critical current density for both methods. The enhance of J_c is greater for the samples obtained by addition and it reaches a maximum (more than two times the un-doped one). This effect is a result of the improvement of the texture, which give a better grain orientation and the higher grain size observed by SEM [21]. The results have shown that better

properties are obtained when potassium is added with a possible substitution at the calcium site instead of strontium site.

For practical application, potassium doping by addition seems a better way to improve superconducting properties.

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