

Synthesis of BiPbSrCaCuO superconductors with substitution of Sr by Ag

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Introduction

The Bi- based superconductor system exhibit three different phases described by a general formula $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_{2n+4}$ related to the value of ($n=1,2$ and 3) which are specified by names ($2201 - T_c \sim 10\text{K}$, $2212 - T_c \sim 80\text{K}$ and $2223 - T_c \sim 110\text{K}$). The desirable phase is the $n=3$ ($\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$) because its higher T_c [1]. It is still very difficult to prepare its pure phase because of its special synthetic condition. The above three phases can be used separately as well as in mixture to form superconductors in various forms. Phase transformation from Bi-2212 to Bi - 2223 often takes place during thermal treatments, this phase transformation mad the thermal processes of BSCCO superconductors very complicated as confirmed by Li et al [2] .

Shortly after the discovery of the Bi- based high temperature superconductor, it was found that partial substitution of Bi by Pb allows a lowering of the sintering temperature by enhancing the formation of the Bi -2223 phase [3,4]. It is believed that pb substitution improves the reaction kinetics via formation of a liquid phase [5,6]. In the BSCCO system, two lead oxide compounds could be in equilibrium with the Bi - 2212 and Bi -2223 phases, namely Ca_2PbO_4 phase and the 3321 phase. The effect of Ca_2PbO_4 phase on the phase formation , the microstructure and properties of Bi-2223/Ag tapes has been investigated extensively [7] .

Dawud [8] investigated the effect of the variation of the nominal composition of the compound $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_{2n+4}$ for different values of (n), and the effect of PbO dopant on these compound. The composition of these samples was analysis by X RD, resistivity measurements and the morphological analysis by SEM .

She prepared two types of samples , the first type prepared without adding PbO, and the second type with PbO doped by 5% and concluded that the doping of BSCCO compound with Pb is essential for the enhancement and the stabilization of the high- T_c phase.

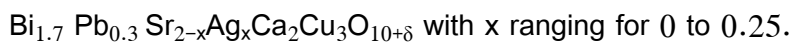
Ozkurt et al [9] prepared $\text{Bi}_{1.7} \text{Pb}_{0.3-x} \text{Nd}_x \text{Sr}_2 \text{Ca}_3\text{Cu}_4\text{O}_{12+y}$ ($0.025 \leq x \leq 0.1$) by melt-quenching method. The effects of Nd substitution on the BSCOO system have been investigated by electrical resistivity, scanning electron microscopy (SEM), x-

ray diffraction (XRD) and magnetic hysteresis measurements. The reported results show that Nd^{3+} doping for Pb^{2+} induces the progressive transformation of the 2223 to the 2212 phase leading to a mixed-phases formation. The R-T results of the sample show two – step resistance transition the first transition occurs at 100K and the second in the range 80–90K, depending on the Nd content. The magnetization decreases with increasing temperature, in agreement with the general behavior of high – T_c materials. The maximum critical current density, J_c value was calculated to be 8.51×10^5 , at 4.2K, J_c decreases with increasing temperature and the substitution level.

Chazala et al. [10] studied the effect of the Ag addition on the superconducting properties of $(\text{Bi}_{0.7} \text{Pb}_{0.3})_2 \text{Ag}_x \text{Sr}_2 \text{Ca}_2 \text{Cu}_3 \text{O}_{10+\delta}$ for $(0 \leq x \leq 0.5)$. They found that disappearance of high transition (108 K) for Ag content $0.2 \leq x \leq 0.4$, indicating that the higher T_c 2223 – type phase transforms to the lower T_c 2212 – type phase. At higher Ag content ($x=0.5$) favor again the formation of 2223 phase Ghazala et al conclude that Ag dose not deteriorate the superconducting properties of the $(\text{Bi}_{0.7} \text{Pb}_{0.3})_2 \text{Ag}_x \text{Sr}_2 \text{Ca}_2 \text{Cu}_3 \text{O}_{10+\delta}$ system but plays a crucial role in the stabilization of the different phases coexisting in the Bi – based superconductors.

This suggests the possibility to use Ag as a stabilizer in the fabrication of superconducting wires, but also points out the need of properly control the Ag content , to prevent unwanted phase transformation and ,thus, the T_c reduction .

In this paper, we have investigated the effect of Ag doping in the Bi –2223 superconductor by preparing a series of sample with complete stoichiometry



Experimental Part

The nominal composition chosen for the present sample was $\text{Bi}_{1.7} \text{Pb}_{0.3} \text{Sr}_{2-x} \text{Ag}_x \text{Ca}_2 \text{Cu}_3 \text{O}_{10+\delta}$. BPSCCO samples were prepared by a conventional solid state reaction method. High purity powders of Bi_2O_3 , $\text{Sr}(\text{NO}_3)_2$, PbO , CaO , CuO and AgO were mixed in stoichiometric proportion calcinated at 830°C for 24 hr and sintered at 860°C for 160hr with rate of 60°C/hr in a tube furnace and the cooled to room temperature by the same rate of heating. 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 : $\text{Cu}_{K\alpha}$,voltage : 40kv , current : 20 mA, wavelength : 1.5405 \AA (

Result and discussions

The resistivity as a function of temperature is one of the most important characteristics of a material as well as to provide an indication of T_c .

Fig. (1) shows the resistive transition behavior of the samples $\text{Bi}_{1.7} \text{Pb}_{0.3} \text{Sr}_{2-x} \text{Ag}_x \text{Ca}_2 \text{Cu}_3 \text{O}_{10+\delta}$ with $x=0, 0.05$ and 0.1 . All the samples show metallic behavior in the normal state and superconducting transition to Zero resistance. It can also be seen from these curves that, the normal state resistivity regularly increases from ($x=0$) to $x=0.1$ with $x=0$. With $x=0$ shows a behavior typical of undoped Bi-2223: a critical transition temperature (T_c) at 122K and Zero resistance at 115K (T_{off}).

The situation was improved with samples doped with Ag, which helps in inducing or enhancing superconducting in a sample (at $x=0.05$ and 0.1). From fig. (1) and Table (1), we can note that the best sample synthesized was with a nominal composition $\text{Bi}_{1.7} \text{Pb}_{0.3} \text{Sr}_{1.9} \text{Ag}_{0.1} \text{Ca}_2 \text{Cu}_3 \text{O}_{10+\delta}$ with a T_c value equal to 128K.

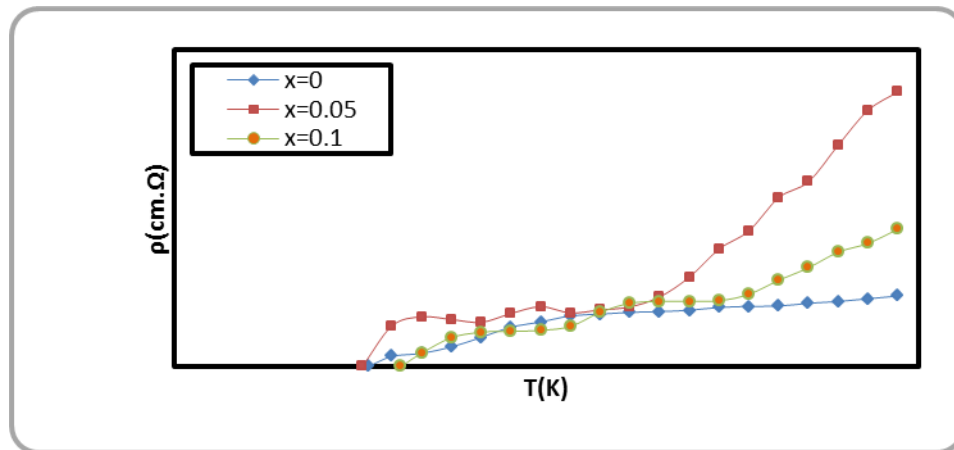


Fig. (1): Temperature dependence of resistivity for $\text{Bi}_{1.7} \text{Pb}_{0.3} \text{Sr}_{2-x} \text{Ag}_x \text{Ca}_2 \text{Cu}_3 \text{O}_{10+\delta}$ system with $x=0, x=0.05$ and $x=0.1$.

More increases of Ag to 0.15 and 0.2 decrease the transition temperature to 125K and 99K respectively as shown in fig. (2). Remarkably, a further Ag addition $X=0.25$ converts the sample to 2223 phase as clearly shown by the re-appearance of the transition temperature at ~ 110 K.

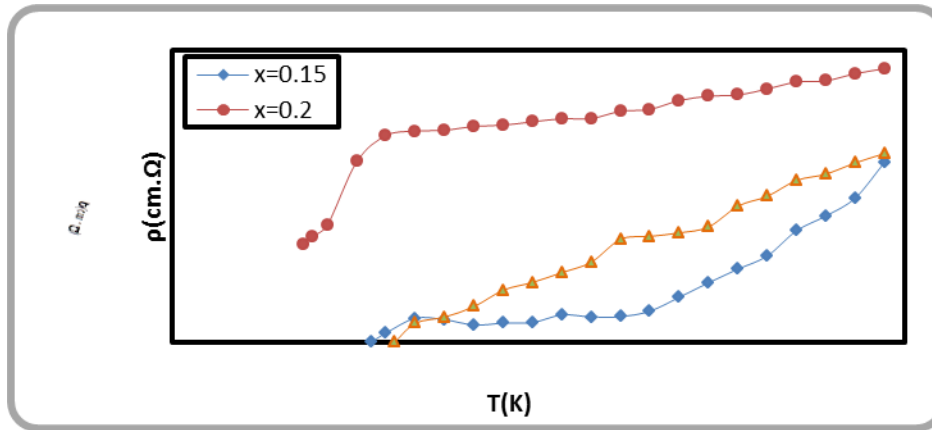


Fig. (2): Temperature dependence of resistivity for $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_{2-x}\text{Ag}_x\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ system with $x=0.15$, $x=0.2$ and $x=0.25$.

This result indicates that the Ag has catalytic effect on the reaction to form the high – T_c phase within the x value equal to 0.05 and 0.1. However, certain amount of Ag is necessary for the occurrence of this reaction, while excessive Ag substitution promotes another reaction, assist the formation of low T_c phase ^[11]

The crystal structure of the prepared samples was studied in the x– ray diffraction

(XRD) analysis. Two kinds of information were obtained from the XRD spectra;

the identification of superconducting phases contained in the samples as well as their relative fractions and the estimation of lattice constants of the corresponding phases .

The lattice parameters a, b and c have been estimated using d – values and (hkl) reflections of observed x–ray diffraction patterns through the software program based on cohen's least square method. It could be seen from the spectra that there are two main phases in all samples ,i.e, high – T_c phases (2223), low – T_c phase (2212) with Miller indices of orthorhombic structure. In addition, there was non superconducting phase which corresponds to the impurity phases CaPbO_2 , Sr-Ca-Cu-O and CuO_2 in agreement with Caskun et al. ^[12] (see fig.3).

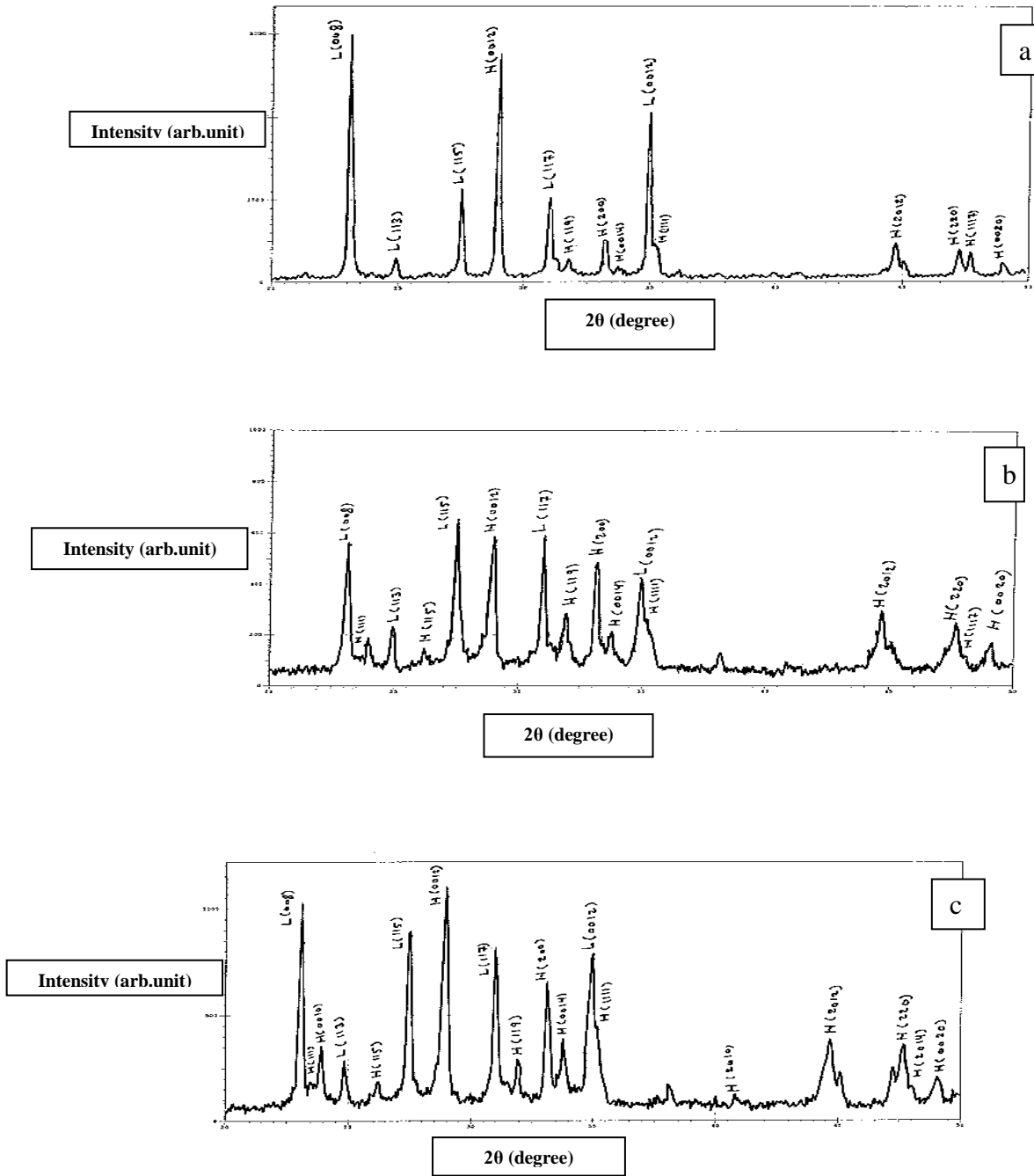


Fig. (3): X-ray diffraction patterns of $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_{2-x}\text{Ag}_x\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ samples (a) $x=0$, (b) $x=0.05$ (c) $x=0.1$. H-HighT_c phase, L-low T_c phase.

It can be noticed from these figures that partial substitution of Ag instead of Sr leads to form a large fraction of high – T_c phase 2223. Thus, the above results suggest that the growth of the high – T_c phase is promoted by Ag –doping , this may be attributed to the ordered growth under the partial melting point and / or Ag substitution for Sr .

Table (1) show an increase in the c–axis lattice constant for Ag–doped samples as comparable with free sample ($x=0$) , the reason is due to the substitution of Ag for Sr where the ionic radii of Ag^{1+} is longer than that of Sr^{2+} which renders c–

Zn	a (Å (b (Å (c (Å (T_c (K)
0	5.3693	5.5390	37.1538	122
0.05	5.3846	5.4072	37.1572	126
0.1	5.4135	5.3857	37.1614	128

parameter to be longer or deformed .

Table (1): Values of lattice constants, and transition temperature of unit cell.

Conclusions:

Our conclusion from the results can be summarized as follows:

1. The substitution of Ag in Sr for the compound $Bi_{1.7} Pb_{0.3} Sr_{2-x} Ag_x Ca_2 Cu_3 O_{10+\delta}$ with $x=0.05$ and 0.1 exhibited a maximum value of T_c found to be 126K and 128K respectively .
2. XRD pattern analyses have shown an orthorhombic structure, and there are at least two superconducting phases.
3. A partial substitution of Sr by Ag ($x=0.05, 0.1$) was found to promote the formation of Bi–2223 phase.

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