

**INFLUENCE OF ADDITION OF Eu_2O_3 NANOPARTICLE ON THE
STRUCTURAL AND SUPERCONDUCTING PROPERTIES OF
 $(\text{Bi}_{1.6}\text{Pb}_{0.4})\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ CERAMICS**

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ABSTRACT

Superconducting ceramics of $(\text{Bi}_{1.6}\text{Pb}_{0.4})\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10} + x \text{Eu}_2\text{O}_3$ were prepared by conventional solid state reaction route. The influence of Eu_2O_3 nanoparticles, added to (Bi, Pb)-2223, ($0.00 \leq x \leq 0.05$) at the final heat treatment processing stages on the structure and superconducting properties were investigated. Bulk specimens were characterized by using X-ray diffraction patterns, four-probe method and scanning electron microscopy (SEM). The addition of Eu_2O_3 caused the growth of the low (Bi, Pb)-2212 superconducting phases in specimens. Large pores together with apparently smaller grain sizes are seen as a consequence of Eu_2O_3 accommodation in BSCCO structure. In the region of the 101 K, the critical temperature T_C gradually decreased with the increase of Eu_2O_3 content.

INTRODUCTION

Since the discovery of Bi-Sr-Ca-Cu-O superconductors, many studies on the structure of these conductors have been carried out [1-3]. The conclusion has consistently suggested that the BSCCO superconductors have three phases. The three phases of superconductors have different critical temperature, the Bi-2201 has 10K, the Bi-2212 has 80K, and the Bi-2223 has 110K. The transition temperature T_C increases with increasing carrier concentration until it passes through a maximum, after which it reduces. Application interest is however concentrated in the Bi-2223 phase with $n=3$, as compared to other phases. It is also expected that a small amount of Eu_2O_3 addition in BSCCO system instead of substitution would enhance connectivity between grains and increase pinning centers between grain boundaries [4].

In the present work, the addition of Eu_2O_3 nanoparticles in (Bi, Pb)-Sr-Ca-Cu-O superconductor is presented. The specimens were prepared by the conventional solid state method and physical characterizations were performed on the specimens. The method used in analyzing data and the results are given in following sections.

MATERIAL AND METHOD

Samples were prepared by the solid state reaction method. The nominal composition of

the samples was $(\text{Bi}_{1.6} \text{Pb}_{0.4})\text{Sr}_2 \text{Ca}_2 \text{Cu}_3 \text{O}_{10}$ (where $x=0.00-0.05$) using high purity chemicals of Bi_2O_3 , PbO , SrCO_3 , CaCO_3 , CuO and Eu_2O_3 nanoparticulate (Aldrich, 99.9%). The powders were mixed in suitable amounts and then pre- calcined at 800°C for 24 h. After first calcinations, the powder was ground and then pressed into pellets with a diameter of 13 mm and thickness of 1 mm under the pressure of 7 tones. Finally, the pellets samples were sintered at 850°C for 120 hours in air then cooled down to room temperature in the furnace. The samples were analyzed by X-ray diffraction method using $\text{Cu K}\alpha$ radiation ($\lambda=1.5418 \text{ \AA}$), to check the phase purity and impurity to determine their structure and lattice parameter. The resistivity of the samples was measured by the standard four-probe method with silver paints as electrical contacts. The specimen morphology was studied by scanning electron microscopy (SEM).

RESULTS AND DISCUSSION

Figure. 1 shows the X-ray diffraction patterns for specimens with various Eu_2O_3 concentrations $x=0.0-0.05$. Most peaks intensities for the specimens with $x=0.005$ and 0.01 are similar and the peaks belong to the Bi-2212 and Bi-2223 phases can easily be identified from the figure. With addition of $x=0.005 \text{ Eu}_2\text{O}_3$ nanoparticulate to pure sample, high(H) and low(L) phases are not different but the low phase peaks belongs to L(008) are increasing and the H(013) disappeared completely by increasing Eu_2O_3 amount. In comparison between two different concentrations, in $x=0.01 \text{ Eu}_2\text{O}_3$ the H(008) intensity is reduced and the L(115) peak intensity is increased. With increasing amount of Eu_2O_3 , a new peak begins to appear at about $2\theta=33.27^\circ$ in the sample with $x=0.03$. In this sample, the peak intensity of all high phases are increased and those belong to the low phase are diminished. Other unidentified phases exist distinctly at $2\theta=22.13^\circ$, 25.36° and 47.22° in Eu_2O_3 nanoparticules-added pure BSCCO. New peaks belonging to Bi-2201 phase was also determined at $2\theta=21.66^\circ$ and 29.77° . At higher 2θ angle, the addition of Eu_2O_3 resulted in the appearance of many high phase peaks. For example H(02 12), H(1119) and H(128) peaks show considerable strength in intensity at their respective positions. In $x=0.03$, it is observed that the intensity of the four main high phase peaks is much prominent. Contrary to the above, the $x=0.05$ added sample shows a considerable decrease in the high phase peak intensity. The volume fraction of present phases in all specimens studied were estimated and presented in Table1. Volume fractions of Bi-2212 and Bi-2223 phases were calculated from peak intensities according to the following equation.

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

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

where I is the intensity of the present phase. As shown in Table 1, the amount of Bi-2223 phase decreases and that of Bi-2212 increases with further addition of Eu_2O_3 nanoparticulate. As observed for Eu_2O_3 addition, sample with $x=0.03$, the volume

percentage of Bi-2223 phase increased [5]. However, other Eu_2O_3 concentrations show less high phase volume favorability. With exception to $x=0.03$, the volume percentage of Bi-2212 is however increased. Lattice parameters a and c were calculated based on the orthorhombic unit cell and presented in Table 1.

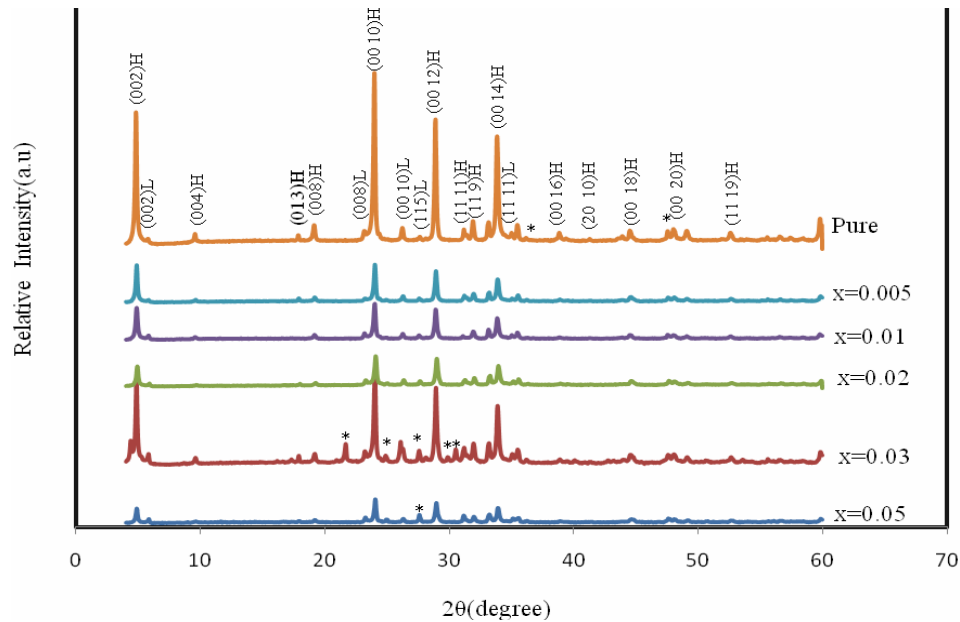


Figure 1: XRD patterns for the (Bi, Pb)-2223, $0.00 \leq x \leq 0.05$ nanoparticulate Eu_2O_3 .

Table 1: Physical data of $(\text{Bi}_{1.6}\text{Pb}_{0.4})\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ specimens Eu_2O_3 .

X	Lattice parameters (Bi-2223) $\text{\AA}$		Volume (%)	
	a	c	Bi-(2223)	Bi- (2212)
0.000	5.4102	37.1293	92.67	3.4
0.005	5.4101	37.1264	83.2	6.2
0.010	5.4100	37.1250	81.5	17
0.020	5.4070	37.1180	91.3	7.9
0.030	5.4107	37.1432	94.3	0.54
0.050	5.0900	36.9800	82.7	6.5

SEM images of the surface (cross-section) fractured from pellets of specimen with $x=0.0-0.01$ were shown in plate 1. Traces of the (0 0 l) peaks, some of which of high intensity, (100%) in the X-ray data confirm the BPSCCO preferred orientation in all specimens. As shown in plate 1, all specimens possess the platelet-like structure of randomly distributed grains. The occurrence of this structure is evidence of (Bi, Pb)-

2223 phase formation from the Bi-2212 matrix assisted mainly by the liquid phase available during heat treatment [6]. As the sintering of the specimen progressed, the PbO-assisted liquid phase composition becomes more homogeneous and eventually lead to the formation of large crystal like (Bi, Pb)-2223 grains. It is reported that high uniaxial compacting pressure during pelletization and the addition of substantial amount of PbO allows a compact arrangement of (Bi, Pb)-2223 grains. In $x=0.01$ and $x=0.02$ specimens, a few complete grains are seen scattered in mostly porous plane. For $x=0.03$ and $x=0.05$ specimens, blocks of layered compact grains with some degree of fusion are seen in the micrograph. A relatively small grain sizes were scattered along the surface plane indicating that the addition of Eu_2O_3 is well accommodated in BSCCO structure. On the other hand, the pure BSCCO sample morphology is seen to consist of platelet grains.

Figure 2 shows the temperature dependence of the electrical resistance for samples with various Eu_2O_3 concentration. The critical temperature T_C gradually decreases with increasing x . These results are consistence with the structural data deduced from X-ray diffractometry shown in figure 1. For specimens with $x=0.02$ and 0.05 however, X-ray data confirm the presence of considerable amount of Bi-2212 phase in their final structure. Normal state resistance is high and both $T_{C(0)}$ and $T_{C(\text{onset})}$ for these specimens are that of the Bi-2212 phase in the specimens with $x>0.02$. Thus, the decrease in $T_{C(0)}$ and $T_{C(\text{onset})}$ are further evidence that the presence of the increasing amounts of Eu_2O_3 nanoparticles in Bi-2223 phase stabilizes and promotes the growth of Bi-2212 phase at the expense of Bi-2223 one [7].

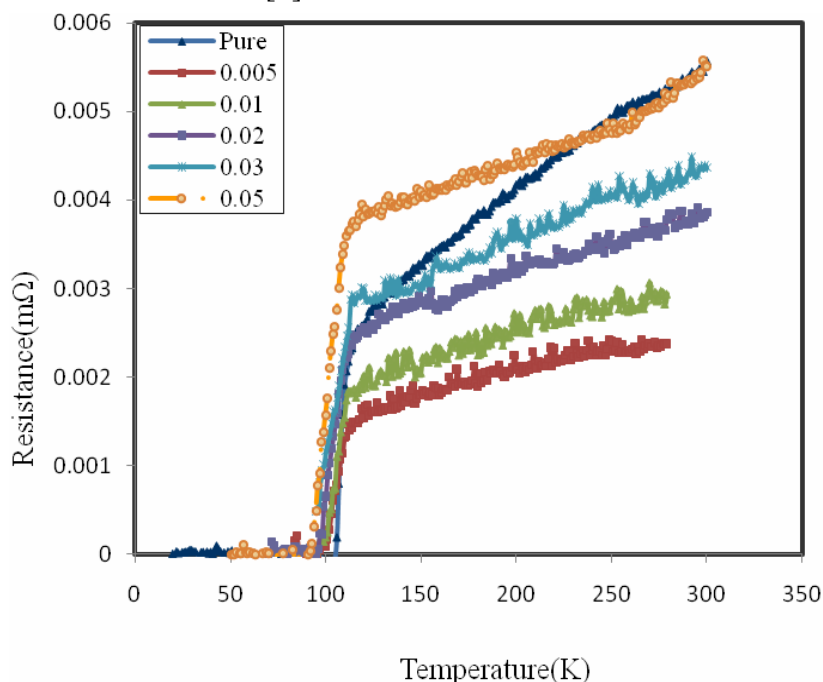


Figure 2: Variation of normalized resistivity as a function of temperature.

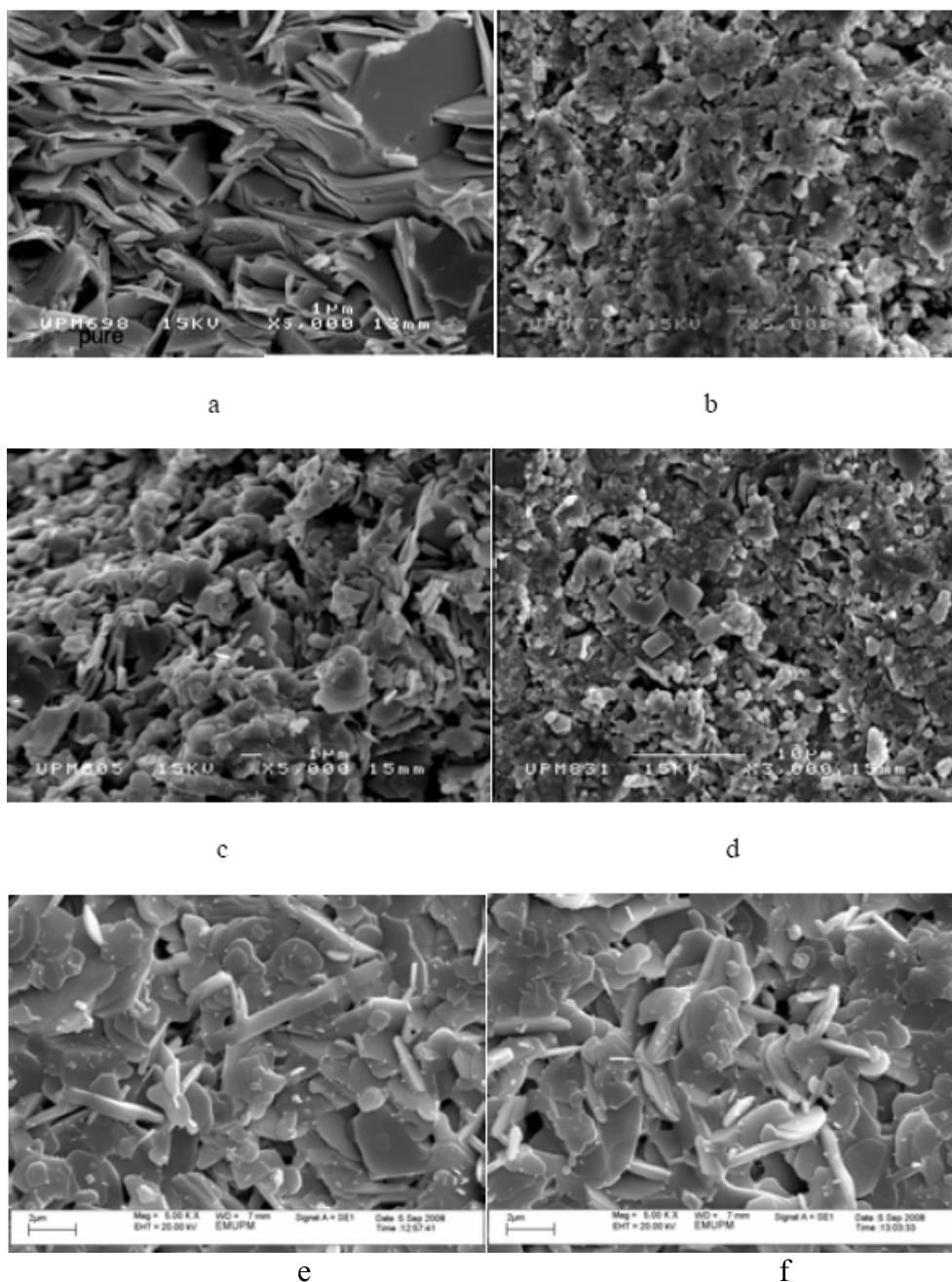


Plate 1: SEM micrographs of the specimens after the final stage of heat treatment, (a) $x=0.00$ (b) $x=0.005$ (c) $x=0.01$ (d) $x=0.02$ (e) $x=0.03$ (f) $x=0.05$

CONCLUSION

A number of $(\text{Bi}_{1.6}\text{Pb}_{0.4})\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10} + x\text{Eu}_2\text{O}_3$ ($x=0.0-0.05$) samples with different x have been prepared. Specimens with about 90% Bi-2223 phase were first prepared by the conventional reaction route in pellet form. These pellets were crushed to a fine powder and then added to a systematic amount of Eu_2O_3 nanoparticles. X-ray

diffraction data shows that the addition of $x=0.03$ of Eu_2O_3 nanoparticles helped in improving the formation of the high phase. This observation is confirmed from SEM and R (T) data. It is very clear to trace the exact effect of the Eu_2O_3 nanoparticles in the structure of the (Bi, Pb)-2223 system from SEM, XRD and R(T) data.

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