

## Physical Properties of Melt-Cast Annealed $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_3\text{Cu}_4\text{O}_{12}$ Compound

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### Abstract

A  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_3\text{Cu}_4\text{O}_{12}$  compound was produced by melt-casting method. The microstructure of the sample was studied by Scanning Electron Microscopy. Phase analysis was carried out by X-ray diffraction patterns and EDAX. The electrical resistance exhibits the existence of a superconducting phase with an onset temperature  $T_c$  at 110 K along with a minor phase with an onset  $T_c$  at 80 K. It has been found that the superconducting phase temperature  $T_c$  increases with increasing sintering temperature.

**Key Words:** H- $T_c$  Superconductivity, BSCCO, Critical Current

### 1. Introduction

After the discovery of La-Ba-Cu oxide superconductors [1], significant research efforts have been reported on the new superconducting materials. Since the observation of superconductivity in BiSrCaCuO by Maeda and co-workers [2], there have been many studies on the preparation and the structural identification of the superconducting phase. The general formula for BiSrCaCuO superconductors is known as  $\text{Bi}_2\text{Sr}_2\text{Ca}_{n-1}\text{Cu}_n\text{O}_{2n+4}$  ( $n=2,3,4$ ) [3,4]. Current researches on the preparation of simple high  $T_c$ -phase superconducting ceramics concentrate on partially replacing Bi in the structure by Pb-doping [5,6]. Pb is mostly used as a dopant in order to enhance the zero resistance transition temperature and volume fraction of the high- $T_c$  phase.

In this study, we have prepared, characterized and investigated the effect of sintering period on the formation of superconductivity in a nominally  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_3\text{Cu}_4\text{O}_{12}$  compound.

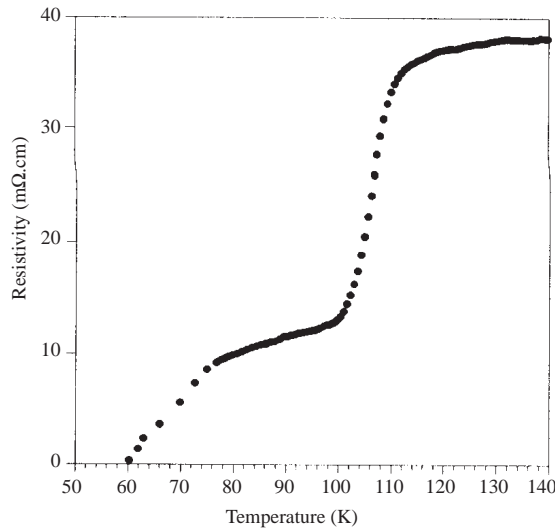
## 2. Experimental

The starting materials  $\text{Bi}_2\text{O}_3$ ,  $\text{SrCO}_3$ ,  $\text{CaO}$ ,  $\text{CuO}$ ,  $\text{PbO}$  were mixed in the stoichiometric values of the nominal compositions. This mixture was calcinated at  $750\text{ }^\circ\text{C}$  for 20 hours in air. The calcinated materials were then ground and placed in a programmable furnace at room temperature. It was suddenly heated within a few seconds to  $1200\text{ }^\circ\text{C}$  in a platinum crucible, and then the melted sample was poured onto a preheated copper mould in order to produce cylindrical bars of diameter 5 mm and length of 10 cm. The cylindrical rods were then sintered at  $840\text{ }^\circ\text{C}$  for 50, 100 and 150 hours in a programmable muffle furnace, in air.

Electrical resistance measurements were carried out by the standard four probe DC method. X-ray diffraction patterns were obtained with a Rigaku D/Max. 3. C diffractometer by using monochromatic  $\text{CuK}\alpha$  radiation in the  $2\theta = 3^\circ\text{-}60^\circ$  range. SEM photographs for the study of the microstructure were taken by using a JEOL. JSM. 6400.

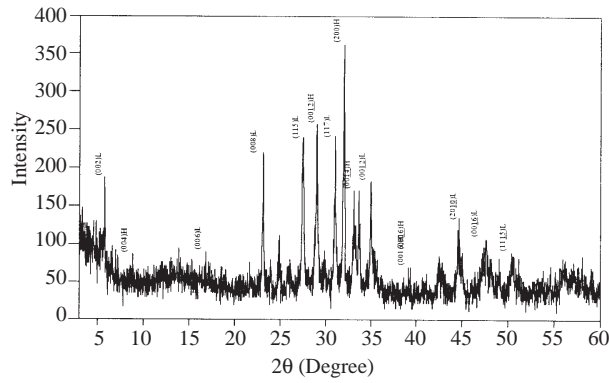
## 3. Result and Discussion

The resistivity-temperature plots for the sample sintered for 150 h is given in Fig. 1. The highest value of resistivity is about  $38\text{ m}\Omega\text{-cm}$  at temperature of 140 K. It then starts gradually decreasing down to 110 K which can be explained by the metallic character of the sample. Two onset temperatures observed at the temperatures of  $\sim 110\text{ K}$  and  $\sim 80\text{ K}$ , respectively, are most likely due to the presence of both the high -  $T_c$  and low -  $T_c$  phases in the sample. Transition  $\Delta T$  is about 30 K and resistivity is zero at 60 K. This indicates that the high- $T_c$  phase is not the dominant phase in the sample.

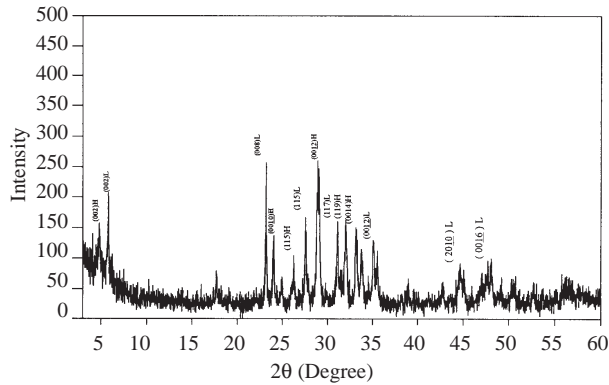


**Figure 1.** Resistivity temperature variation for the sample  $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_3\text{Cu}_4\text{O}_{12}$  sintered for 150 h.

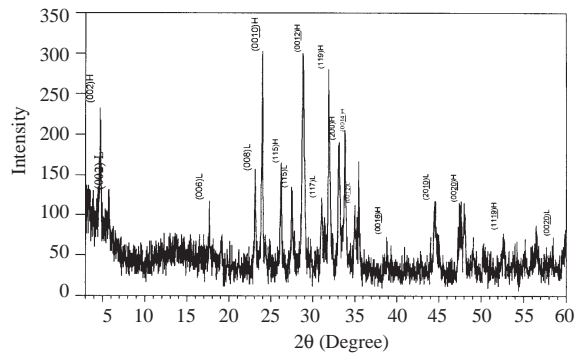
The XRD diffraction patterns of 50 h, 100 h, 150 h sintered samples are shown in Fig. 2. It was found that the samples of 2234 rods prepared by melt-cast-anneal method show not only the high- $T_c$  phase (2223), which contain low- $T_c$  phase (2212), but also non-superconducting phases (2201, Sr-Ca-Cu-O and CaO). The dominant phase in the high- $T_c$  phase corresponds to a transition temperature of 110 K. The existence of secondary phases after 150 h annealing at 838 °C can be attributed to the nominal composition chosen and the preparation technique. The indication of increased high  $T_c$  phase at  $2\theta = 4.7^\circ$  at peak profile and a decreased low- $T_c$  phase  $2\theta = 5.2^\circ$  is clearly seen in Fig. 3. It was concluded that the volume fraction of the 2223 phase increases by prolonged annealing [7]. The lattice parameters of 150 h annealed sample were calculated to be  $a = 5.29 \text{ \AA}$  and  $c = 37.14 \text{ \AA}$  which correspond to the tetragonal microstructure of BSCCO sample.



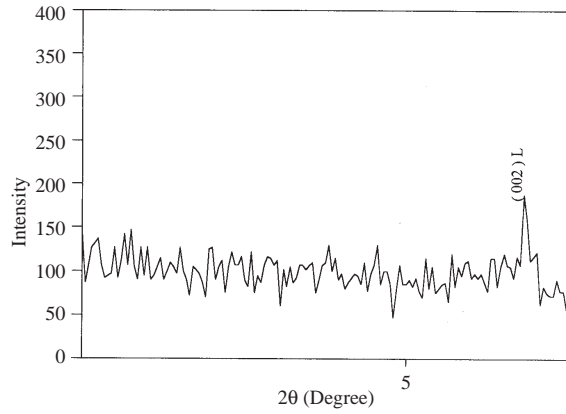
**Figure 2a.** X-ray diffraction patterns for the sample sintered 50 h.



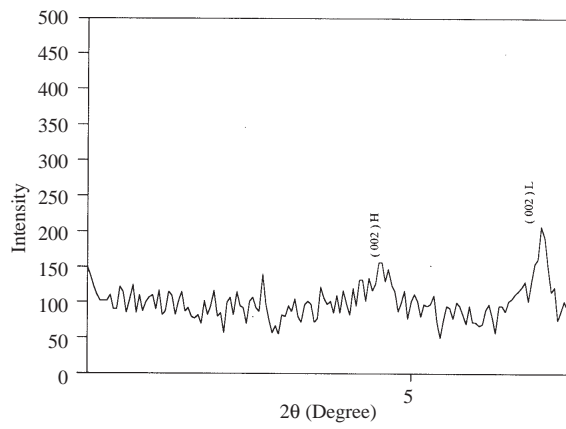
**Figure 2b.** X-ray diffraction patterns for the sample sintered 100 h.



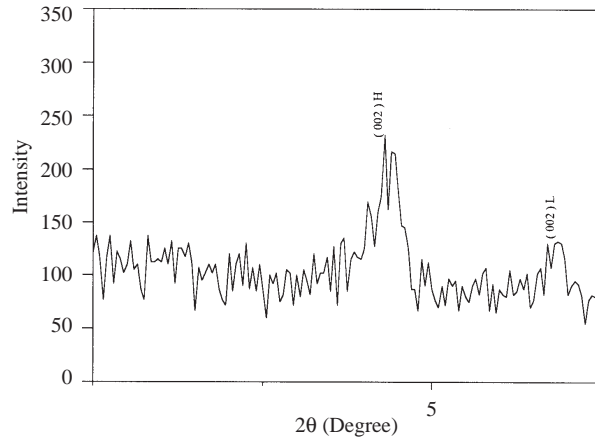
**Figure 2c.** X-ray diffraction patterns for the sample sintered 150 h.



**Figure 3a.** X-ray diffraction patterns between  $2\theta = 3^\circ - 6^\circ$  sintered for 50 h.



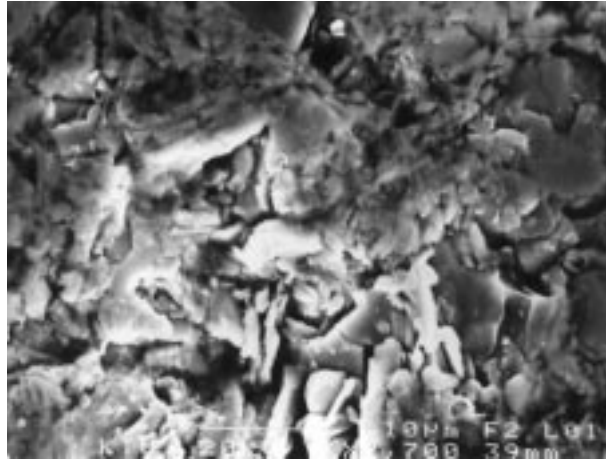
**Figure 3b.** X-ray diffraction patterns between  $2\theta = 3^\circ - 6^\circ$  sintered for 100 h.



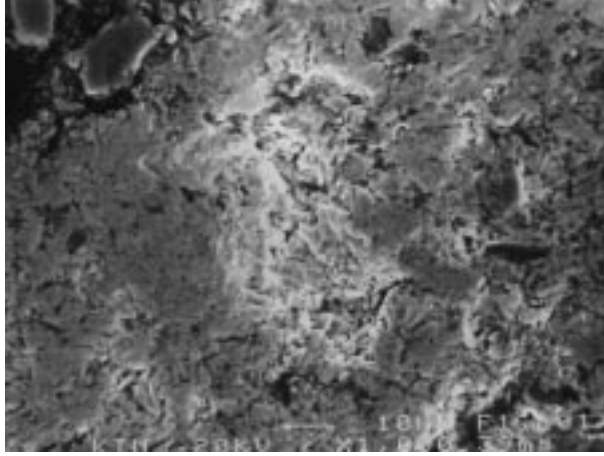
**Figure 3c.** X-ray diffraction patterns between  $2\theta = 3^\circ - 6^\circ$  sintered for 150 h.

EDAX has been used to analyze the content of Bi, Pb, Sr, Ca in the grains of the sample. According to the results, while the content of Ca and Sr elements decrease, the content of Bi increases with increasing temperatures. So it can be concluded that Pb cations in the grains have replaced some Sr cations rather than Bi cations.

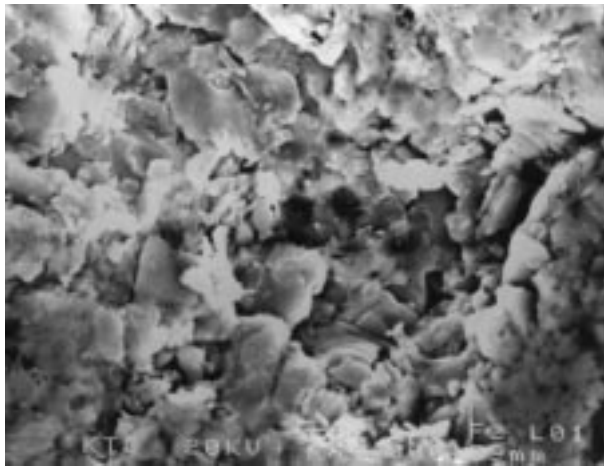
The SEM photographs are illustrated in Figs. 4, for different sintering times. In Fig. 4a and 4b, there are both needle shapes and grain structure but, in Fig. 4c, there is only grain. Among these is a distributed porous structure which caused small critical currents. The critical current has been determined by using the standard four-probe method the liquid Nitrogen temperature and is of the order  $10.7 \text{ A/cm}^2$  for 100 hours, and  $21.2 \text{ A/cm}^2$  for 150 hours sintering times, respectively.



**Figure 4a.** SEM photograph of the sample sintered 50 h.



**Figure 4b.** SEM photograph of the sample sintered 100 h.



**Figure 4c.** SEM photograph of the sample sintered 150 h.

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