

Effect of Sintering Time On Sb Added BiPbSrCaCuO Superconducting Ceramics

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Abstract

In this study, superconducting Sb doped BiPbSrCaCuO ceramics with a fixed nominal composition were prepared and sintered at 850°C for different periods. The structural phases of the sintered ceramics were determined by means of the measurements of the resistivity-temperature behaviour and of the XRD analysis. Sb doping along with Pb doping in the BiSrCaCuO ceramics was observed to increase the volume fraction of the high T_c phase, the onset temperature and the critical temperature T_c . The critical temperature for the 100 hour sintered sample was 4K higher than that of the 60 hour sintered sample.

Introduction

Since Maeda et al. [1] discovered superconductivity in the BiSrCaCuO (BSCCO) system, significant research has been focused on the synthesis and the use of various dopants to increase the T_c of these ceramics in addition to increasing the volume fraction of the high T_c phase in multiphase ceramics. It has been shown that substituting Bi partially with Pb enhances the formation of the high T_c BSCCO phase [2]. Current research indicates that ceramics which are a mixture of low and high T_c phases are formed by doping with Pb or Pb and Sb together.

Sb doping alone in the $Bi_{2-x}Sb_xSr_2Ca_2Cu_3O_y$ ceramics was found to have a 120°K superconducting onset temperature and the resistivity was zero at 92K with the formation of a new superconducting phase by Hangbao et al [3]. They later determined a T_c of 132K for $Bi_{1.9}Pb_xSb_{0.1}Ca_2Cu_3O_y$ ceramics prepared by the conventional ceramic method. The possibility of the existance of a new phase with a T_c of 140K along with the 85K low T_c and 110K high T_c phase was also reported in that work which, was the highest T_c reported in the BSCCO system up to that moment [4]. The onset temperature was found to be 140K with zero resistivity being obtained at 92K for Pb+Sb doped ceramics in another study which also reported the formation of a monoclinic phase along with the

high T_c (2223) phase [5]. Kijima et al. [6] reported an increase in T_c by 4K on Sb doping of the Pb doped BSCCO ceramics.

Chandrachood et al [7] reported a T_c of 132K for $Bi_{1.9}Sb_{0.1}Sr_2Ca_2Cu_3O_y$ ceramics without Pb doping. Sb doping was, on the other hand, concluded to lower the Cu^{+3} concentration and unit cell parameters which may decrease the T_c [8]. In yet another study it is stated that Sb controls the oxygen stoichiometry which was considered to be very important and a T_c of 130K was reported for Sb doped ceramics [9].

$Bi_{1.5}Pb_{0.4}Sb_{0.1}Sr_2Ca_2Cu_3O_y$ ceramics prepared by a melt-quenching technique was determined to have a T_c of 140K and this phase appeared along with the formation of a glassy state [10]. In another melt-quenching study no evidence of a new superconducting phase with a T_c of around 130K was found in similar ceramics. However, Sb doping was reported to improve the superconducting properties of the material [11].

In our previous work, the formation of a monoclinic phase and a low T_c phase with a T_c of 80K was reported for $x=0.4$, Pb-free $Bi_{2-x}Sb_xSr_2Ca_2Cu_3O_y$ ceramics [12]. The effect of Sb doping on the T_c of Pb doped BSCCO ceramics is not yet fully understood. In this study, $Bi_{1.5}Pb_{0.4}Sb_{0.1}Sr_2Ca_2Cu_3O_y$ ceramics were prepared by conventional ceramic method. Pelletized samples were heat treated at a fixed temperature for various periods. Sintered ceramics were further characterized and the presence of the recently claimed 140K high T_c phase was investigated.

Experimental Procedure

The Sb and Pb doped BSCCO ceramics were prepared by the conventional ceramic method. Predetermined amounts of high purity (99.99% Aldrich) starting chemicals (Bi_2O_3 , PbO , $SrCO_3$, Sb_2O_3 , $CaCO_3$, CuO) were used for the preparation of the ceramics of fixed nominal composition of $Bi_{1.5}Pb_{0.5}Sb_{0.1}Sr_2Ca_2Cu_3O_y$. These powders were well mixed and ground by using a mortar and pestle and it was further calcined at 800°C for 25 hours in a (Carbolite 1400 Furnace). Calcined powders were ground again to form a fine powder. Pellets 13 mm in diameter and 1 mm in thickness were pressed by uniaxial compaction in a die at 500 MPa. These pellets were sintered at 850°C for 60, 80 and 100 hours in air and then furnace cooled to room temperature. Resistance-temperature data were obtained by using four point probe DC method. XRD pattern were obtained by a Rigaku D-Max 3C-PRD in the $2\theta=0-50^\circ$ range. SEM micrographs were taken by using a JEOL-JF-JXA 733 SEM.

Results and Discussion

The temperature dependence of the electrical resistivities of the $Bi_{1.5}Pb_{0.4}Sb_{0.1}Sr_2Ca_2Cu_3O_y$ ceramics sintered at 850°C for 60, 80 and 100 hours (Samples A,B,C, respectively) is given in Figure 1. The onset temperature for all samples is approximately 132K. This is about 15K higher than our previously published onset temperature for Pb doped BSCCO ceramics. The resistivity-temperature curve is linear up to the onset temperature, in accordance with the metallic character of the samples. Zero electrical resistivity

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is obtained in a single step at 108, 110, and 112K, respectively, for samples A to C. The transition width decreases with increasing sintering time which is indicative of better phase homogeneity. Sb doping of the Pb doped BSCCO ceramics increases the T_c 3-4K in our work [13] and a similar result was also observed by others [7]. It is possible to say that the volume fraction of the high T_c phase increases with increasing sintering time and the high T_c phase appears to be the dominant phase in these ceramics.

The XRD patterns of the samples are shown in Figure 2. All the peaks were identified by using the tabulated data of D. Pandey et al. [14] and S. Bansal et al. [15].

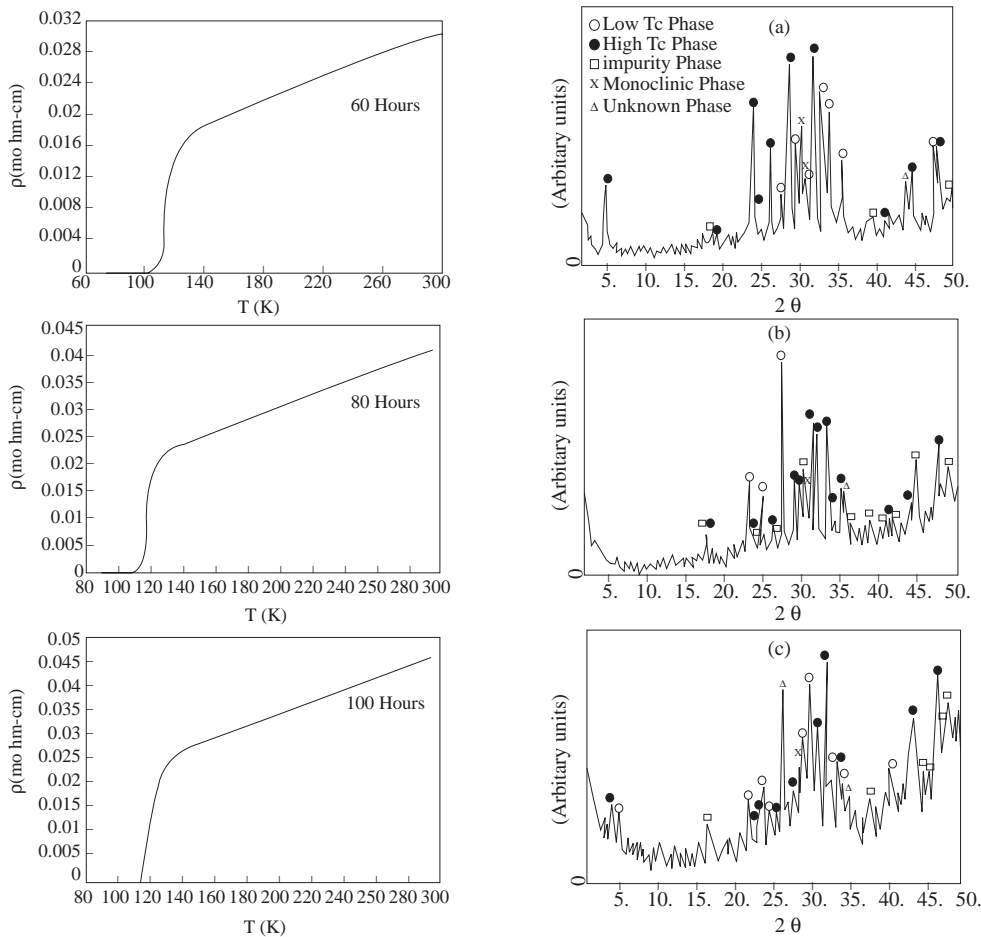


Figure 1. Temperature dependence of electrical resistivity for the $Bi_{1.5}Pb_{0.4}Sb_{0.1}Sr_2Ca_2Cu_3O_y$ ceramics at $850^\circ C$ for 60 h, 80 h and 100h.

Figure 2. X-ray diffraction patterns of samples sintered at $850^\circ C$ a) 60 h b) 80 h c) 100 h

All the samples appear to have a multiphase structure which indicates the difficulty in obtaining a single phase material by the conventional ceramic method. Single phase ceramics were obtained by melt-quenching technique and under controlled atmospheres at shorter sintering periods [10,11]. An analysis of the number of corresponding peaks and their intensities indicates that the high T_c phase is the dominant phase in all three samples in this work. The commonly observed Ca_2PbO_4 impurity phase at $2\Theta=17.8^\circ$ was also detected in all samples. Few peaks for CuO, Ca_2CuO_2 , and Ca_2PbO_4 impurity phases in the $2\Theta=30-50^\circ$ range were detected but the number of these peaks and their intensities decreased with longer sintering time.

The most intense peak in XRD pattern of sample C belongs to the high T_c phase which also indicates increase in the volume fraction of the high T_c phase with sintering time. The intense low T_c phase peaks in the $2\Theta=30-35^\circ$ range in sample A can not be further detected in sample C. Two monoclinic phase peaks ($d=2.95.3A$) were detected in all samples which was previously identified as non-superconducting phase by Qinlun Xu et al. [5]. These peaks were observed to be more intense peaks in our earlier Pb-free Sb doped BSCCO samples [12].

References

- [1] H. Maeda, Y. Tanaka, M. Fukutomi and T. Asono, Jpn.J.Appl.Phys, 27 (1988) L209.
- [2] M. Takano, J. Takada, K. Oda, H. Katigauchi, Y. Mīura, Y. Ikeda, Y. Tomi and H. Mazaki, Ibid. 27 (1988) L1041.
- [3] L. Hongbao, E. Xiaonong, C. Yaozu, R. Yaozhong, C. Zhaojiia and E. Yuheng, Physica C, 156 (1988) 804.
- [4] L. Hongbao, C. Liezhao, Z. Ling, M. Zhiqiang, L. Xiaoxian, Y. Zhidong, X. Bai, M. Xianglei, Z. Guien, R. Yaozhong Solid State Communacations, 69 (1989) 867.
- [5] Qinlun XU, Z. Chen, G. Meng and D. Peng, Jpn. J. Appl. Phys, 29 (1990) 1918.
- [6] N. Kijima and R. Gronsky, Jpn. J. Appl. Phys, 31 (1992) 182.
- [7] M.R. Chandrachood. I.S. Mulla, A.P.B. Sinha Appl. Phys. Lett, 55 (1989) 1472.
- [8] S.X. Dou. H.M. Liu, N.X. Tan, Y.J. Sheng and W.K.Jones. Physica C, 158 (1989) 97.
- [9] S.A. Agnihotry, S. Chandra, P.P. Bakare and S.K. Date, Physica C, 212 (1993) 381.
- [10] J.A.A. Khan, N.M. Butt, A. Mahmood, R. Shaheen, G. Bashir, K.A. Shadid, Journal of Materials Science, 27 (1992) 6321
- [11] T. Komatsu, R. Sato, K. Imal, K. Matusiate and T. Yamashira, Übid, 27 (1988) L533.

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- [12] K. Kocabaş, Turkish Journal of Physics, 18 (1994) 415.
- [13] K. Kocabaş and N. Kazancı, Turkish Journal of Physics, 18 (1994) 541.
- [14] D. Pandey et al. Physica C, 173 (1991) 476.
- [15] S. Bansal et al. Physica C, 173 (1991) 260.