



Synthesis and enhancement of current density in cerium doped Bi(Pb)Sr(Ba)-2 2 2 3 high T_c superconductor

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ABSTRACT

Bismuth based superconductors (BSCCO) having 2 2 2 3 phase is highly carried by the researchers to study the structural and other properties. The effect of substitution of cerium element on calcium site of bismuth based Bi(Pb)Sr(Ba)-2 2 2 3 high T_c superconductor for the enhancement of superconducting properties like critical temperature (T_c) and critical current density (J_c) are studied. The nominal compositions of $\text{Bi}_{1.6}\text{Pb}_{0.4}(\text{Sr}_{1-y}\text{Ba}_y)_2(\text{Ca}_{1-x}\text{Ce}_x)_2\text{Cu}_3\text{O}_y$ (where $x = 0.0, 0.1, 0.2, 0.4$ and $y = 0.0, 0.4$) ceramic superconductor were prepared by wet chemical method. Stoichiometric amounts of Bi_2O_3 , PbO , $\text{Sr}(\text{NO}_3)_2$, BaCO_3 , CaCO_3 , CuO and CeO_2 were used as starting materials. The effect of substitution of cerium on calcium site and its effect on current density of Bi-2 2 2 3 phase were studied. To study the structural morphology and surface analysis X-ray diffraction (XRD) and scanning electron microscopy (SEM) was done. It is observed that with the substitution of cerium on calcium site not only the phase pure material is obtained but also the current density increases.

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1. Introduction

The high- T_c ceramic superconductors, based on the number of CuO_2 planes in the typical multilayer blocks can be classified into single layer material, e.g., $\text{Bi}_2\text{Sr}_2\text{CuO}_{6+\delta}$ Bi-(2 2 0 1) and bi-layer materials e.g., $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$ Bi-(2 2 1 2) and tri-layer materials, e.g., $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10+\delta}$ Bi-(2 2 2 3) [1,2]. This structural characteristic has a direct association with the superconducting properties. In this family, the superconducting phase transition temperature (T_{c0}) increases with the layer number (n) for $n = 3$ and then starts to decrease [3]. Out of these three phases, 2 2 2 3 phase has always been of interest because of its highest T_c (~ 110 K), atmospheric stability, and ability to be rolled into long wires or tapes [4,5]. Even though various experimental and theoretical efforts have been done, a decisive microscopic understanding of this evolution has not yet been reached. It has been reported by various groups [6] that Pb at Bi site up to an optimum concentration of 0.3–0.4 has proven useful in enhancing (2 2 2 3) superconducting phase and thus facilitating its purity. Substitution of lead [7] with other elements is being attempted for further improvement in these materials especially if these could enhance the critical current density (J_c). The critical current density is a very important property of a

superconductor for technological applications [8]. Large critical current densities (J_c) are required. In addition to an increase of the proportion of Bi-2 2 2 3 phase during processing, a highly aligned Bi-2 2 2 3 microstructure is essential for a high J_c value because a low J_c value usually results in a grain boundary weak-coupling problem as well as a random growth habit of the superconducting grains [9]. The current transport in granular superconductors is governed not only by the nature of the grains but also interconnects between them. It is believed that processing methods enhance the critical current density by improving the grain boundary stoichiometry, the inter-granular coupling and the grain alignment [10]. The conventional solid state reaction method is the most employed technique to produce ceramic superconductors in which the starting materials usually includes oxides, carbonates and other salts, which are mixed, homogenized and heat treated at specific temperature [11]. This method usually includes many disadvantages such as large particle size, long heat treatment and several intermediate grindings therefore it is time consuming and has the tendency to produce contaminated products [12]. These problems can be overcome by wet chemical methods [13]. The superconducting BSCCO material prepared with the sol-gel method has better physical and chemical properties in comparison with the conventional solid state reaction method [14]. The powder obtained by this technique has smaller grain size, better homogeneity and high purity. This is due to the initial mixture of cations on an atomic scale in solution so as to enhance the reaction. It is well known that the intergranular

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critical current density value of Bi-2 2 2 3 superconducting materials depends on many factors like temperature gradient, chemical composition, crystallite size, heat treatment, pelletization pressure, alignment of grains, etc. [15,16]. The superconducting samples having higher critical current density are much useful in production of large magnetic fields like in particle accelerators and in the medicine field such as magnetic resonance imaging (MRI) and many electrical power appliances where energy losses can be reduced with the help of these materials. Superconductors in the large scale field are today limited to niche applications: medical devices (MRI and NMR spectrometer) and “big science” (high energy physics and thermonuclear fusion). The biggest SC machines (LHC and forthcoming ITER) are in Europe (Geneva) [17].

The rare-earth elements have different ionic radii and so have great impact as substitute for studying physical properties of high T_c superconductors [16]. The main aim of the present study is to investigate the effect of cerium substitution on calcium site of Bi(Pb)Sr(Ba)(2 2 2 3) phase.

2. Experimental procedure

Wet chemical method (sol-gel method) was used for the sample preparation with nominal composition $\text{Bi}_{1.6}\text{Pb}_{0.4}(\text{Sr}_{1-y}\text{Ba}_y)_2(\text{Ca}_{1-x}\text{Ce}_x)_2\text{Cu}_3\text{O}_y$ (where $x=0.0, 0.1, 0.2, 0.4$ and $y=0.0, 0.4$). The precursors were weighed on the digital balance according to their stoichiometric ratios. Precursors having 99.9% purity used were bismuth oxide (Bi_2O_3), lead oxide (PbO), strontium carbonate (SrCO_3), barium carbonate (BaCO_3), calcium carbonate (CaCO_3), cerium oxide (CeO_2) and copper oxide (CuO). Each precursor was then dissolved in the nitric acid. One molar solution of each precursor was prepared by adding the suitable amount of distilled water. These solutions were then poured into the solution of ethylene glycol under continuous stirring and heating at 100°C . After an hour, a light blue gel was formed which was then fired till it was turned into black powder at the same temperature. This powder was then ground in agate mortar and pestle. This grinded powder was then placed into heating furnace for 10 h for calcination step at 600°C . After the calcination powders were pressed into pellets at 70 lb/m^2 . The prepared pellets were in cylindrical disk shape having diameters and thickness of 13 mm and 3 mm respectively. These disks were then sintered at specific temperatures for further characterization and compositions were named respectively as S1 = $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$, S2 = $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_{1.6}\text{Ba}_{0.4}\text{Ca}_2\text{Cu}_3\text{O}_y$, S3 = $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_{1.6}\text{Ba}_{0.4}\text{Ca}_{1.8}\text{Ce}_{0.2}\text{Cu}_3\text{O}_y$, S4 = $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_{1.6}\text{Ba}_{0.4}\text{Ca}_{1.6}\text{Ce}_{0.4}\text{Cu}_3\text{O}_y$, and S5 = $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_{1.6}\text{Ba}_{0.4}\text{Ca}_{1.2}\text{Ce}_{0.8}\text{Cu}_3\text{O}_y$.

3. Results and discussion

In order to study the superconducting properties, the crystal structure of the samples was determined by the X-ray diffraction (XRD) technique. The obtained patterns from XRD were indexed and the samples were analyzed for their electrical properties.

3.1. X-ray diffraction analysis

X-ray diffractographs of the samples, after final sintering were taken. These studies gave the information about crystal structure, lattice constants and the type of the unit cell. Patterns were taken by PANalytical X-ray diffractometer using $\text{Cu-K}\alpha$ radiation (wavelength $\lambda = 1.5406 \text{ \AA}$) with a step size of 0.02. The diffraction data was taken at room temperature since there is no change in the lattice constant and the structure of high- T_c superconductors above and below the transition temperature [18].

Table 1

Lattice parameters of the samples obtained from XRD analysis. Crystal structure for all the samples is orthorhombic.

Composition	Lattice constants		
	a (Å)	b (Å)	c (Å)
$\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$	5.44 (4)	5.50 (4)	36.93 (5)
$\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_{1.6}\text{Ba}_{0.4}\text{Ca}_2\text{Cu}_3\text{O}_y$	5.42 (4)	5.37 (3)	37.12 (5)
$\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_{1.6}\text{Ba}_{0.4}\text{Ca}_{1.8}\text{Ce}_{0.2}\text{Cu}_3\text{O}_y$	5.40 (4)	5.41 (4)	36.72 (4)
$\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_{1.6}\text{Ba}_{0.4}\text{Ca}_{1.6}\text{Ce}_{0.4}\text{Cu}_3\text{O}_y$	5.42(4)	5.43 (5)	36.55 (4)
$\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_{1.6}\text{Ba}_{0.4}\text{Ca}_{1.2}\text{Ce}_{0.8}\text{Cu}_3\text{O}_y$	5.43 (4)	5.41 (4)	36.53 (3)

For all the samples X-ray diffraction patterns are given in Fig. 1, and the indexed peaks are shown. The patterns show that the samples are superconducting containing only high- T_c 2 2 2 3 phase. Only sample S1 shows a few peaks of low- T_c phase with no other impurities peaks.

The volume fractions of high- T_c and low- T_c phase can be estimated from the intensities of high- T_c , low- T_c phase peaks [2] by the following formulae:

$$\text{Bi-(2 2 2 3)\%} = \left[\frac{\Sigma I(2 2 2 3)}{\Sigma I(2 2 2 3) + \Sigma I(2 2 1 2)} \right] * 100 \quad (1)$$

$$\text{Bi-(2 2 1 2)\%} = \left[\frac{\Sigma I(2 2 1 2)}{\Sigma I(2 2 2 3) + \Sigma I(2 2 1 2)} \right] * 100 \quad (2)$$

where I is the peak intensity of the phases present. The proportion of Bi-2 2 2 3 was 71.04% and Bi-2 2 1 2 was 28.95% respectively in sample S1. This shows that lead substitution on the bismuth site favors the kinetics of formation of high- T_c phase. All the other compositions were with the pure high- T_c Bi-2 2 2 3 phase. The lattice parameters were calculated from (hkl) values of the indexed peaks. Lattice parameters are given in Table 1.

3.2. Scanning electron microscopy

The surface morphology of the cerium added superconducting samples was studied by scanning electron micrographs (SEM). The surface micrographs of the prepared samples are shown in Fig. 2. It is observed that with the addition of cerium the morphology of the samples changed very interestingly to flaky plate like structures. Superconducting grains are seen to be connected with each other strongly. This morphology is expected to be helpful for the enhancement of current density.

3.3. DC electrical resistivity measurements

The experiment was performed by lowering the temperature slowly from its room value towards the boiling point of liquid nitrogen (77 K). Resistivity measurements were taken after final sintering step. The maximum value of $T_{c,0}$ observed for the samples and the sintering times are given in Table 2. The resistivity versus temperature graphs is shown in Fig. 3.

Table 2

DC electrical measurements of all the prepared samples.

Composition	$T_{c,0} (\pm 1)\text{K}$	Sintering time (h)
(S1) $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$	107	72
(S2) $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_{1.6}\text{Ba}_{0.4}\text{Ca}_2\text{Cu}_3\text{O}_y$	112	60
(S3) $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_{1.6}\text{Ba}_{0.4}\text{Ca}_{1.8}\text{Ce}_{0.2}\text{Cu}_3\text{O}_y$	108	90
(S4) $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_{1.6}\text{Ba}_{0.4}\text{Ca}_{1.6}\text{Ce}_{0.4}\text{Cu}_3\text{O}_y$	110	150
(S5) $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_{1.6}\text{Ba}_{0.4}\text{Ca}_{1.2}\text{Ce}_{0.8}\text{Cu}_3\text{O}_y$	113	150

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