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# Substitution of Sm at Ca site in $Bi_{1.6}Pb_{0.4}Sr_2Ca_{2-x}Sm_xCu_3O_y$ superconductors

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#### Abstract

We have investigated the effect of the partial substitution of Ca by Sm in the Bi-2223 superconducting samples prepared by standard solid-state reaction method. Our investigations consisted of DC electrical resistivity, AC susceptibility, X-ray diffraction (XRD) and scanning electron microscopy (SEM) measurements. We measured the critical transition temperatures, activation energies and irreversibility lines from the resistivity versus temperature curves under DC magnetic fields in the range of 0 and 0.6 T. The superconducting transition temperature,  $T_c$ , and activation energy,  $U_0$ , were found to decrease with increasing Sm concentration and with increasing applied magnetic field. Increasing the Sm content shifted the irreversibility temperature to lower values. The AC susceptibility measurements were carried out at 80 A/m field strength and f = 211 Hz frequency. XRD patterns and SEM micrographs are given to provide information about Bi-2223 and Bi-2212 phases and their grain size. The possible reasons for the observed degradation in microstructural and superconducting properties due to Sm substitution were discussed. © 2007 Elsevier B.V. All rights reserved.

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#### 1. Introduction

It is well known that superconductivity is suppressed by the presence of magnetic ions in the conventional metallic superconductors. This behaviour can be understood in terms of the pair-breaking mechanism [1]. In high- $T_c$ superconductors, it is well known that the superconducting properties are related to the chemical dopings, preparation conditions and hole concentrations. The objectives of doping works are to optimize the hole concentration, to introduce pinning centres, to enhance the formation of Bi-2223 phase [2]. Variation in the  $T_c$ ,  $J_c$ , and lattice parameters are obtained via doping the system with other elements at different levels and under various preparation conditions [3–11]. Terzioglu et al. [3] have recently investigated the effect of substitution of Sm for Ca on superconducting, microstructure and mechanical properties of  $Bi_{1.6}Pb_{0.4}$   $Sr_2Ca_{2-x}Sm_xCu_3O_y$  [3,4,6]. It was obtained that superconducting and mechanical properties degrade with increasing Sm content. It was also reported that for x = 1.0 and 1.5 the compounds showed semiconducting behaviour, resistivity increased with decreasing temperature.

The irreversibility line can be determined from various measurements such as DC resistivity and AC susceptibility [12]. Muller et al. [13] first reported the irreversibility line in high temperature superconducting ceramics. The measurements of AC susceptibility are commonly used to determine magnetic and superconducting properties of these materials. In particular, the AC susceptibility measurement is useful in distinguishing between inter- and intragrain properties of the specimens. Two peaks,  $T_p$  and  $T_g$ , reflect inter- and intragranular losses in the ceramic superconductors. They can be distinguished from the temperature dependence of the imaginary part of the complex AC susceptibility only

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when high AC fields were used in the measurement [14,15]. The first peak appears at a temperature  $T_g$  slightly below  $T_c$  and indicates a maximum hysteresis loss for the motion of intragranular loss [15]. The second loss peak appears at a temperature  $T_p$  lower than  $T_g$  caused by the motion of integranular vortices [16]. They both depend on the sample's composition.

In this work, we report X-ray diffraction (XRD), scanning electron microscopy (SEM), magnetoresistivity, and AC susceptibility measurements on Sm substituted  $Bi_{1.6}Pb_{0.4}$  Sr<sub>2</sub>(Ca<sub>2-x</sub>)Sm<sub>x</sub>Cu<sub>3</sub>O<sub>y</sub> samples. We also investigated the effect of the partial substitution of Ca by Sm on the activation energy, the irreversibility line (or temperature), and inter- and intragrain properties of Bi-2223 superconductor.

#### 2. Experimental details

The Sm substituted Bi<sub>1.6</sub>Pb<sub>0.4</sub> Sr<sub>2</sub>(Ca<sub>2-x</sub>)Sm<sub>x</sub>Cu<sub>3</sub>O<sub>y</sub> samples with x = 0, 0.0005, 0.001 and 0.005 were prepared by the standard solid-state reaction method using high purity chemicals Bi<sub>2</sub>O<sub>3</sub> (99.99%), PbO (99.9 + %), SrCO<sub>3</sub> (99.9 + %), CaCO<sub>3</sub> (99 + %), CuO(99 + %) and Gd<sub>2</sub>O<sub>3</sub> (99 + %) [3]. Rectangular bars were cut from the sintered samples for electrical resistivity and AC susceptibility measurements. The typical sample size was (2.9 × 2.1× 12.1) mm<sup>3</sup>. The calcinations and annealing processes of the samples were carried out using a programmable tube furnace from PROTHERM (Model PTF 12/75/200). The samples annealed at 860 °C for 200 h with different Sm substitutions in Bi<sub>1.6</sub>Pb<sub>0.4</sub> Sr<sub>2</sub>(Ca<sub>2-x</sub>)Sm<sub>x</sub>Cu<sub>3</sub>O<sub>y</sub> (x = 0, 0.0005, 0.001 and 0.005) will be hereafter denoted as B0, B1, B2, and B3, respectively.

We measured temperature dependence of resistivity of the samples using usual four-probe method with 5 mA DC current in a cryostat. Magnetoresistivity measurements were made under different DC magnetic fields (0, 0.3 and 0.6 T). The magnetic field was applied parallel to the current direction, by an electromagnet. The transition temperature  $T_{\rm c}$  was determined as the temperature at which zero resistivity was achieved. The reversibility temperature was determined as zero resistance temperature at various selected magnetic fields. The effective activation energy serves as an important parameter for the description of the flux dynamics in the mixed state for high- $T_c$ superconductors. A commonly accepted method for probing the magnetic field dependence of the effective activation energy is to measure the resistive transition in various applied magnetic fields. More accurate values of activation energy,  $U_0$ , can be obtained from transport rather than magnetic measurements due to the widening of the magnetic transition. We have calculated  $U_0$  in this study using the line pinning model [17] by doing approximations from resistivity measurements [18-22] via an Arrheniustype method. We have assumed that the resistivity  $\rho$  has following dependence:

where  $k_{\rm B}$  is Boltzmann's constant. When we plot  $\ln \rho$  versus 1/T curve, the slope of the low resistivity part gives the activation energy  $U_0$ .

Magnetic susceptibility as a function of temperature (75–120 K) measurements were performed using a 7130 AC susceptometer of Lake Shore with a closed cycle refrigerator at 211 Hz frequency and 80 A/m field strength. The values of the  $T_c^{\text{onset}}$  and  $T_p$  were estimated from the real and imaginary parts of AC susceptibility curves, respectively. We could observe only one peak in each of the  $\chi''$  versus T plots. The values of intergranular critical current densities  $J_c(T_p)$  in our samples were calculated using Bean's model from the acquired data.

XRD data were collected using a Rigaku D/Max-IIIC diffractometer with  $CuK_{\alpha}$  radiation in the range  $2\Theta = 4-60^{\circ}$  with a scan speed of 3° per minute and a step increment of 0.02° at room temperature. The lattice parameter *c* was determined from (002), (008), (0,10), and (0014) peaks. The accuracy in determining the lattice parameter *c* was  $\pm 0.001$  Å.

The surface morphologies of the pure and Sm substituted samples were studied by using a JEOL JST-6400 scanning electron microscope.

## 3. Results and discussion

### 3.1. Electrical resistivity and activation energy

We performed the electrical resistivity as a function of temperature between 77 and 130 K to investigate the effect of Sm substitution on the superconducting properties of the Bi<sub>1.6</sub>Pb<sub>0.4</sub>Sr<sub>2</sub>(Ca<sub>2-x</sub>Sm<sub>x</sub>)Cu<sub>3</sub>O<sub>y</sub> samples with  $0 \le x \le 0.005$ . Fig. 1 displays the temperature variation of the normalized resistance (that each sample's resistance normalized at 130 K) of the pure sample (B0) as well as Sm added samples (B1, B2, and B3). Zero-resistivity transition temperatures of the B0, B1, B2 and B3 samples are



$$\rho(T) = \rho_0 \exp(-U_0/k_{\rm B}T),$$

(1)

Fig. 1. Temperature dependence of normalized resistance for the samples.

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