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Research paper

Experimental and theoretical approaches for magnetic, superconducting and structural characterization of $Bi_{1.75}Pb_{0.25}Sr_2Ca_2Cu_{3-x}Sn_xO_{10+y}$ glass ceramics

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ABSTRACT

In this work, the effect of the partial substitution of Sn for Cu on structural, electrical and magnetic properties in the superconducting ceramic glass $Bi_{1.75}Pb_{0.25}Sr_2Ca_2Cu_{3-x}Sn_xO_{10+y}$ system (x = 0, 0.1, 0.5) has been examined. The structural characterization of samples produced by glass-ceramic technique were done by X-ray diffraction (XRD) and scanning electron microscopy (SEM) measurements. The electrical and magnetic properties were determined by resistance-temperature (R-T) measurement and magnetization-magnetic field measurement (M-H) in the low temperature by using vibrating sample magnetometer (VSM), respectively. From the results of R-T measurement, the value of critical temperature (T_c^{offset}) has been decreased from 98 K to 73 K with increasing Sn- concentration. The critical current density has also been decreased with increasing magnetic field intensity. It has been observed from M-H plots that it was decreased with increasing the value of temperature. We have observed from M-H curves that the area size of the hysteresis curve decreases with increasing the value of temperature.

1. Introduction

Glass ceramics are materials that are produced by controlled crystallization procedure, consisting nucleation and crystal growth stages, of appropriate glass. It is possible to produce non-porous materials containing fine-grained and regularly dispersed crystals in the microstructure of glass produced amorphously by suitably selecting the conditions of the heat treatment and composition of the glass. For this reason, there are many glass ceramic products developed for different engineering applications [1–4]. One of these application areas is glass ceramic superconductors. Many researchers are investigating the effects of doping different elements in glass ceramic superconductors [5,6]. The purpose of these investigations is to understand the basic mechanism of high temperature superconductivity and to improve superconducting properties (T_c, J_c, I_c etc.) of materials. The most studied system of glass ceramic superconductors is the BiSrCaCuO (BSCCO) superconductor. Superconductivity in BSCCO systems was first discovered in 1988 by Maeda et al. [7]. Subsequent work has shown that the properties of these type of superconductors are very sensitive to initial compositions, preparation methods and variable parameters of the selected method (sintering temperature and sintering time, etc.). In BSCCO systems, there are three phases which can be obtained by the general formula $Bi_2Sr_2Ca_{n-1}Cu_nO_{2n+4+y}$. In the general equation, n gives the number of Cu-O layers in a unit cell. For n = 1, $Bi_2Sr_2CuO_{6+y}$ (Bi-2201) with a transition temperature of 30 K; for n = 2, $Bi_2Sr_2CaCu_2O_{8+y}$ (Bi-2212) with a transition temperature of about 85 K, for n = 3, $Bi_2Sr_2Ca_2Cu_3O_{10+y}$ (Bi-2223) with a transition temperature of 110 K phases are obtained [8].

In this study, Sn was doped instead of Cu in $Bi_{1.75}Pb_{0.25}Sr_2Ca_2Cu_{3-x}Sn_xO_{10+y}$ structure and doping effect on structural, superconducting and magnetic properties of the materials were investigated. The reason for the preference of Sn doping is that the Sn atoms are closer to Cu atom in terms of electronegativity, lattice parameters and bond lengths. In addition, Sn doping has not been investigated before in place of Cu in the glass ceramic BSCCO system. In the literature, calcium (Ca) was replaced with Sn in the BSCCO system and it has been found that the critical temperature decreases with the addition [9,10].

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2. Experimental method

In this study, high purity Bi_2O_3, $SrCO_3$, $CaCO_3$, CuO, SnO and PbO compounds were mixed at appropriate stoichiometric ratios for 1 h in an agate mortar to prepare a glass sample in $Bi_{1.75}Pb_{0.25}Sr_2Ca_2Cu_{3-x}Sn_xO_{10+y}$ composition using the glass ceramic method. The obtained powder mixture was put into the alumina crucible and allowed to stand in the oven for 90 min at about 1150 °C to allow the powder to be melted. The melt obtained in the crucible was taken out of the furnace and very rapidly poured onto the pre-cooled copper plate and pressed with another copper plate which was cooled in the same way. After this process, about 1–2 mm thickness of black glossy glasses were obtained. These processes are carried out in a fast and cold environment, thus preventing regional crystallization. The same process was done for different Sn additive ratios. These glasses were placed in a tubular furnace initially at room temperature and waited for 120 h at 840 °C. The samples obtained were named x = 0, 0.1 and 0.5, depending on the addition ratios.

The electrical properties of the heat treated samples for determining the superconductivity transition temperatures were studied by measuring resistance as a function of temperature. Electrical and magnetic measurements of the materials were performed with Cryogenic Q-3398 brand Vibrating Sample Magnetometer (VSM) system. The four-point method was used for resistance measurements and contacts were made with copper wires using silver paste on solid samples of approximately 3 mm × 5 mm. In the R-T measurements, the temperature of the system was determined by applying the dc current of 1 mA on the sample after reaching the thermal balance at the relevant value during heating.

In magnetization-field (M-H) measurement, the change of magnetization in the material is obtained as a function of the magnetic field by starting from a maximum value such as 9 T, descending to the negative equal field value and then returning to the starting field value. M-H measurements were made at 20, 40 and 60 K constant temperature values between −9 and +9 T field values. The critical current (J_c) values obtained from the M-H curves are calculated by using the Bean formula. The weights of samples for measurements specified and results in terms of emu/g were obtained.

The phase formation and the lattice parameters of the samples are obtained using Bruker D8 Advance diffractometer with CuK_α radiation range between 2θ = 5°–70° with the scanning speed 2 °/min at room temperature. Surface morphology, grain connectivity and grain size of samples are investigated by Leo EVO-40 VPX scanning electron microscopy (SEM).

3. Results and discussions

One of the important analyzes in determining the electrical properties of a superconducting material is R-T measurement. As is known, pure superconducting materials have zero resistance at certain temperature values. Since materials with different elements such as BSCCO cannot obtain a single phase precisely, the temperature at which the resistance of the material is zero can be a temperature range (ΔT_c), not a single value. In this case, the temperature at which the resistance of the cooled material begins to fall to zero is represented by T_c^{onset} and the temperature at which it is completely zero is represented by T_c^{offset}. Fig. 1 shows the resistance measurements for the samples $Bi_{1.75}Pb_{0.25}Sr_2Ca_2Cu_{3-x}Sn_xO_{10+y}$ (x = 0, 0.1 and 0.5), which have been subjected to heat treatment at 840 °C, depending on the temperature. The obtained data are given in Table 1.

In Fig. 1, it can be said that all samples show metallic properties from room temperature to about 113 K, superconducting structures are formed near the zero resistance value and they are completely superconducting. The difference between Sn-doped and non-doped samples is shown in Table 1. Sn-Cu substitution did not cause a significant change in the critical temperature (T_c^{onset}) of the materials, but significant decrease in T_c^{offset} values was observed. The

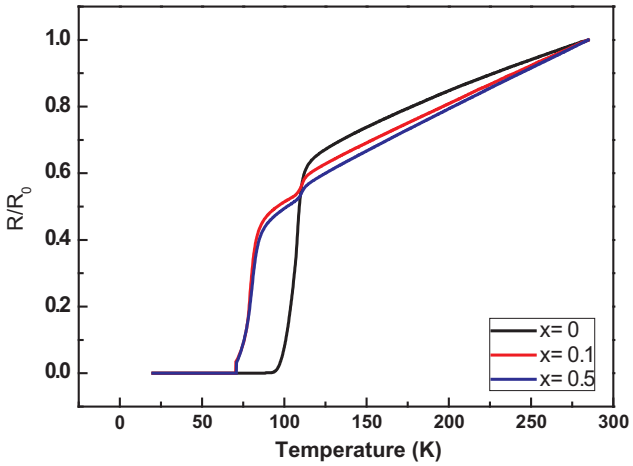

Fig. 1. R/R_0-T graph for $Bi_{1.75}Pb_{0.25}Sr_2Ca_2Cu_{3-x}Sn_xO_{10+y}$ (x = 0, 0.1 and 0.5) samples.

Table 1
Critical transition temperatures for the samples $Bi_{1.75}Pb_{0.25}Sr_2Ca_2Cu_{3-x}Sn_xO_{10+y}$ (x = 0, 0.1 and 0.5).

Samples	T_c^{onset} (K)	T_c^{offset} (K)	ΔT (K)
0	112.38	98.11	14.27
0.1	112.96	73.27	39.69
0.5	112.47	73.09	38.38

$Bi_{1.75}Pb_{0.25}Sr_2Ca_2Cu_3O_{10+y}$ sample (x = 0) became superconductor at 112.38 K and was completely superconducting at 98.11 K.

From the electrical measurement results, the resistance fluctuation that occurs in samples x = 0, 0.1 and 0.5 can be expressed as the increase of Bi-2212 and impurity phases expressed in our previous work [11]. Correspondingly, it was observed that the critical temperature range (ΔT_c) increased with the Sn displacement. It is also thought that the reduction in the critical temperature is caused by the defects in the crystal structure occurred during Sn-Cu substitution [12–14].

In our previous structural analysis study [11], it was observed that the impurity ratio in the crystal structure increased with the amount of Sn and the peak intensities of Bi-2223 structure decreased with Cu and Sn partial displacement. Here, Bi-2212 phase, which is the superconducting low temperature phase, increased with Bi-2223 phase decrease. It is seen that with the addition of Sn, $SrSnO_3$ (022-1442-ICDD), Ca_2PbO_4 (003-0131-ICDD) and $Ca_{1.2}Sr_{0.8}PbO_4$ (048-1525-ICDD) impurity peaks are formed in the structure and the intensity of these peaks is increased due to Sn ratio [11]. The increase in the peak intensities of the Bi-2212 phase and the decrease in the peak intensities of the Bi-2223 phase with Sn doping are shown in Fig. 2a and b, respectively.

In Fig. 3, SEM images of the samples doped at 0.0 and 0.5 were given. From these images, it is seen that the structure of undoped sample is more irregular than that of 0.5, the highest doped, sample. In addition, it was observed that the grain size increased and the gaps between grains decreased with the addition. In this case, it can be said that crystallization, grain contact and homogeneity increased with the addition [11].

The magnetization (M-H) measurements of the produced samples were carried out at constant temperatures of 20, 40 and 60 K, with a sweep speed of 200 Oe/s and under a magnetic field between −9 T and +9 T. Before each measurement, the samples were cooled to related temperature values after heating up to 100 K without magnetic field (ZFC), for zero trapped magnetic field in the material. At these temperatures, magnetization measurements were performed after the samples were brought to equilibrium with a sensitivity of 0.05 K. At each stage of the measurements, the magnetic field was changed at a rate of 5 mT/s. The magnetic field was applied parallel to the c axis. In