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STRUCTURAL AND ELECTRICAL PROPERTIES OF HIGH AND LOW-DENSITY Yb-DOPED Bi(Pb)-2223 SUPERCONDUCTOR

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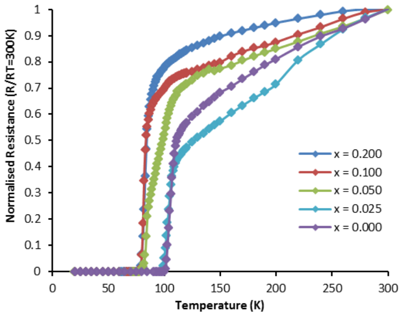
Graphical abstract



Abstract

Ytterbium (Yb)-doped of Bi(Pb)-2223 with varying concentration was prepared by co-precipitation (COP) and solid state reaction (SSR) to produce high density and low density samples respectively. In this work, the samples were characterized by X-ray diffraction analysis (XRD) and resistivity measurement system. Substitution of Yb in Bi(Pb)-2223 decreased the volume percentage of 2223 phase, the length of c-parameter, T_C and J_C towards higher concentration of Yb. The crystallographic structure is tetragonal in a low concentration of Yb but changes to orthorhombic at higher Yb-doped. The high-density samples have a higher volume percentage of 2223 phase and critical temperature, T_C for the same concentration of Yb compared to low-density samples. However, the critical current density, J_C in low-density samples is higher compared to high-density samples due to the large surface area in a porous structure made by sucrose. The large surface area favors improving the grains connectivity during the sintering process.

Keyword: Superconductor, BSCCO, yb-doped

1.0 INTRODUCTION

A crucial part to obtain good superconducting materials is preparation technique. Many researchers have developed various techniques such as solid state reaction, sol-gel, co-precipitation, infiltration, etc. Each technique has advantages and disadvantages. Solid state reaction technique, for example, is the simplest and cheapest technique to prepare superconducting materials. However, this technique needs long sintering time and produce large grains size compared to sol-gel and co-precipitation. Among all of these techniques, co-precipitation is suitable to produce the finest grains and, therefore, increase the critical temperature, TC and critical current density, JC. Both characteristics are influenced by the amount of carrier concentration, charge localization, interlayer coupling effects, interplay, the coexistence of high-temperature superconductivity with the magnetic order, the formation of pseudogaps and fluctuating stripe phase [1]. Substitution with other elements such as metal and rare earth onto the YBCO and BSCCO were found to improve the superconductivity. A small amount of dopant would enhance the superconductivity but would cause detrimental effects if over-doped [2]. It causes the increment of either the electron or the hole concentration in the structure due to the dopant ion. The holes are delocalized and are free to migrate from the location of dopant. This migration begins once the hole concentration in the charge-reservoir region reaches some critical value and both the onset of superconductivity and increase in the TC are supposed to be accompanied by increased transfer of holes from the charge reservoir to the cuprate planes [3].

Substitutions of various elements with different concentration at certain sites and adjusting preparation method affect phase formation and physical properties of BSCCO system. Therefore, the studies of substitution are important to optimize the hole concentration, to introduce pinning centers and to enhance the formation of Bi-2223 phase. The rare earth substitution such as Y, Nd and Dy in Ca site of Bi2Sr2Ca2Cu3Oδ gave significant effects to the carrier concentration on the superconducting properties [4-6]. Other studies reported that substitution of Y, Lu and Er at the Ca site on Bi(Pb)-2223 superconductor was found to induce additional of Bi-2212 and Bi-2201 phases [7]. Khan *et al*. reported that substitution of Ca2+ by Yb3+ in Bi1.7Pb0.3Sr2Ca2-xYbxCu3Oy degraded the superconducting properties and decreased the hole carrier concentration [8].

In this paper, we have investigated the effects of Yb-doped in Ca site of Bi2Sr2Ca2Cu3Oδ superconducting system prepared via co-precipitation (COP) to produce high density samples and conventional solid state reaction to produce low density samples. Sucrose was added at a certain ratio with BSCCO powder to get low-density samples.

2.0 EXPERIMENTAL

Two preparation methods were used to prepare Bi1.6Pb0.4Sr2Ca2-xYbxCu3Oy where x = 0.000, 0.025, 0.050, 0.100 and 0.200 samples i.e. co-precipitation (COP) and solid state reaction (SSR). High-density samples were prepared using COP while low-density samples were prepared using SSR.

2.1 Co-Precipitation Method (COP)

Solution A was prepared by mixing Bi(CH3COO)3), Pb(CH3COO)2 · 3H2O, Sr(CH3COO)2, Ca(CH3COO)2 · xH2O, Cu(CH3COO)2, Yb(CH3COO)3 · xH2O with 300 mL acetic acid. The oxalic acid was dissolved in water: isopropanol (1:1.5) to have a concentration of 0.5 M (solution B). Solution A was added to the stirred solution B in an ice bath and a uniform, stable, blue suspension was obtained. The slurry was filtered followed by the drying stage at 60 °C overnight. Then the powder was subjected to pre-calcination of 12 hrs at 730 °C in air. The pre-calcined powder was reground in an agate mortar for 10 min and calcined at 845 °C in air for 24 hrs. The powders were reground and pressed into pellets with 30 MPa pressure before being sintered at 850 °C for 48 hrs and slowly cooled to room temperature at 2 °C/min [9].

2.2 Solid State Reaction Method (SSR)

The high purity oxide and powders of Bi2O3, PbO, SrCO3, CaCO3, CuO and Yb2O3 were accurately weighed, mixed and wet milled with absolute ethanol in an alumina pot overnight. The mixture was dried out in an oven for 6 hours at a temperature of 120°C to obtain a grey powder. The resulting mixed powder were ground using an agate mortar and pestle for 10 minutes and pre-calcined followed by calcination process in a box furnace at 800°C for 15 hours for each process and intermediate grinding was done at each stage of calcination. The calcined materials were reground, mixed with 0.05g polycrystalline sucrose, C12H22O11 and pelletized using a cylindrical die of 12 mm diameter under a force of 30 MPa. The process of burning the polycrystalline sucrose was done at 400°C for 2 hours. The resulting low-density pellet then sintered for 48 hours at a temperature of 850°C for phase formation contribution.

2.3 Characterization Technique

The critical temperature, TC and the critical current density, JC were measured using resistivity measurement system. The structural properties such as the length of the lattice parameter crystallographic structure were determined using X-Ray Diffractograph.

3.0 RESULTS AND DISCUSSION

The volume fractions of Bi-2223 and Bi-2212 (%) for a high density of the Yb substituted samples prepared via COP obtained from XRD pattern are shown in Table 1. It can be deduced that further substitution of Yb decreased the volume fraction of Bi-2223 phase and increased the volume fraction of Bi-2212 phase from 54.88 % to 23.07 % volume fraction of Bi-2223 for x = 0.025 to x = 0.200. The results of decreasing Bi-2223 phase in this works are consistent with the result in the literature by Khan *et al*. (1999) [10]. Therefore, the substituted Yb has affected on decomposing the structure of Bi-2223 phase and enhanced the Bi-2212 phase.

Table 1 Lattice Parameters, Volume Unit Cell and Volume Fraction of High-density Yb-doped Bi-2223 Samples Prepared via COP

Yb content (x)	Lattice parameters (Å)			Volume of unit cell (V) (Å^3)	Volume fraction (%)	
	a	*b*	*c*		Bi-2223	Bi-2212
0.000	5.4033	5.4033	37.130	1084.0	78.13	21.87
0.025	5.4033	5.4033	37.087	1082.8	54.88	45.12
0.050	5.5686	5.2211	37.071	1077.8	31.01	68.99
0.100	5.6605	5.2049	37.083	1092.6	28.87	71.13
0.200	5.3874	5.4066	37.083	1080.1	23.07	76.93

The length of lattice parameter implies that Yb substitutions changed the crystalline structure from tetragonal to orthorhombic. This behavior is confirmed by previous reports when rare-earth elements were substituted in the Bi-2223 system [11]. It was observed that c-parameter and volume of unit cell of Yb substitution decrease significantly until x = 0.025 Yb concentration but further substitution of Yb causes the trend to becomes ambiguous due to the presence of Bi-2212 phase in the samples. This observation is in agreement with earlier reports concerning Nd substitution for the Ca-site in the Bi-2223 [11].

The previous report suggested that the superconductivity of the oxide superconductor is closely related to the lattice c-parameter. The calculated values of c-parameter and the volume of unit cell decreased significantly until x = 0.050 Yb concentration but further substitution of Yb caused the trend to becomes ambiguous due to the presence of Bi-2212 phase in the samples. Similar behavior has been observed in the previous research involving of rare-earth elements at the Ca-site in the Bi(Pb)-2223 [11].

Table 2 Lattice Parameters, Volume Unit Cell and Volume Fraction of Low-density Yb-doped Bi-2223 Samples Prepared via SSR

Yb content (x)	Lattice parameters (Å)			Volume of unit cell (V) (Å^3)	Volume fraction (%)	
	a	*b*	*c*		Bi-2223	Bi-2212
0.000	5.3939	5.3986	37.131	1081.2	78.57	21.43
0.025	5.3681	5.3651	37.325	1075.8	64.90	35.09
0.050	5.3810	5.3856	37.011	1072.6	58.07	41.93
0.100	5.4018	5.3922	30.614	891.71	36.65	63.26
0.200	5.4264	5.4191	30.540	898.06	27.37	72.63

The summarized data of lattice parameter, volume of unit cell and volume fraction of low-density Yb-doped samples prepared via SSR is tabulated in Table 2. The crystallographic structure shows that samples with 0.000 ≤ x ≤ 0.050 are tetragonal with a lattice length a=b≠c while samples with 0.100 ≤ x ≤ 0.200 are orthorhombic where a≠b≠c. The significant change in the lattice parameters with change in Yb doping level implies that Yb enters into the crystal structure. The Bi-2223 volume fraction in low-density samples is better especially for Yb-doped samples compared with high-density samples even though COP yield very fine grains.

The decrease in the length of c-parameter can be attributed to the fact that the Yb doping leads to an increase in oxygen content in the crystal structure. The excess oxygen is taken up by the Bi-O double planes, causing a tighter binding, which reduce the length of c-parameter. Another reason could be due to the incorporation of substitution ions into the interstitial sites in the unit cell rather than occupy the Ca sites. The substitution ions will cause the distortion between the slabs of the Bi-2223 leading to the formation of Bi-2212 [9, 12]. The replacement divalent cation Ca2+ by trivalent Yb3+ ion reveals the behavior of lattice parameter in the structure. It was believed that the charge neutrality is established by incorporating excess oxygen ions in the bismuth oxide layer of the structure, and the net positive charge in these planes reduces. These will increase the covalency of Bi-O bonds and hence reduced repulsion of bismuth oxide layers [13]. Therefore, c-parameter reduces and improves the stability of the Bi-2212 phase. This observation is in agreement with earlier reports for rare-earth substitution of Sm substituted in Bi-2223 system [14,15] and also for Yb substitutions in Bi-2212 [16] and Yb addition into Bi-2212 system [17,18]. However, since parameter a and b are controlled by the length of the in-plane Cu-O bond, it is very difficult to understand the trend of parameter a and b as a result of substitution [19].

In the case of a/b-axes there is a considerable change both a-parameter and b-parameter where it's length increase significantly. The change of a/b-axis is associated with the change in the Cu-O bond length in Cu-O2 planes [20]. Deviation of these values from the ideal structure values reported in the literature suggests the possible formation of a distorted crystal structure.

The normalized resistances at 300K as a function of temperature for high-density and low-density Yb-doped are shown in Figure 1 and Figure 2 respectively. All the doped samples showed a decrement in critical temperature, TC zero. Wu *et al*. (2007) reported that the decreasing of TC is due to the decrease of hole concentration in the Cu-O plane caused by the dopant at Ca2+ site [5]. The substitution of rare earth at Ca site in Bi(Pb)-2223 provide additional electron that in turn decrease the hole carrier concentration leading to decrease of TC and other superconducting properties.

The curve indicates a metallic behavior for all the samples above the onset temperature (TC onset) and shows a superconducting transition to zero resistance. The resistivity curve shows a smooth curvature of one-step transition Bi-2223 phase for pure and x = 0.025 in high-density samples and only for the pure sample in low-density sample. Other Yb-doped samples have TC zero less than 100 K. This is due to the existence of two types superconducting grains, one formed by the Bi-2223 phase and the second by the Bi-2212 phase. For these samples, upon lowering the temperature, the current goes through paths traversing the grains of the Bi-2223 phase coupled together via some weak links and passes the island of the Bi-2212 phase [21]. One the volume fraction of Bi-2223 phase within the samples is sufficient to make this possible; a one-step resistivity is observed even in the samples that contain a rather large amount of the Bi-2212. However, the curve shows an inflection due to the two-step transition caused by the presence of the Bi-2212 phase for 0.050 concentrations of Yb. The two-step transition is an indication of two phases present (Bi-2223 and Bi-2212) that related to a structural phase transformation and weak links in the sample. Furthermore, the resistivity curve shows a smooth curvature that typically Bi-2212 one step transition for substitution of x = 0.100 and 0.200.

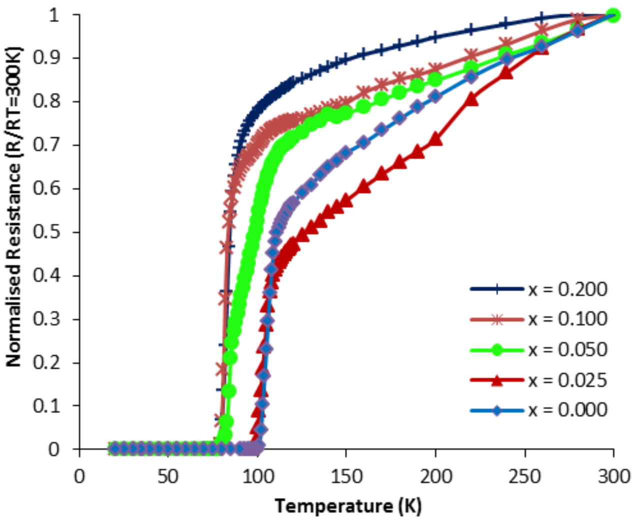


Figure 1 Normalized Resistance At Room Temperature Versus Temperature For High Density Yb-Doped Prepared Via COP

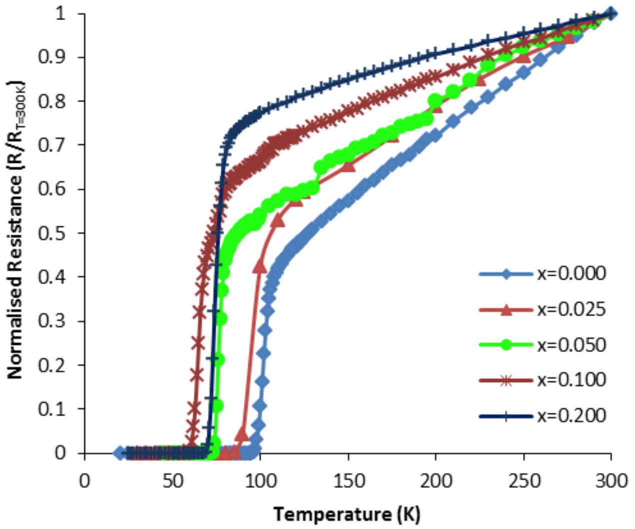


Figure 2 Normalized resistance at room temperature versus temperature for low-density Yb-doped prepared via SSR

Table 3 shows the value of Tc zero, Tc onset, ΔTc and hole concentration of Yb-doped for high and low-density samples. The high-density samples reveal a better result compared to the low-density samples. These results suggest that COP method is better than SSR method due to very fine grains produced by COP method. All the critical temperature, TC decreased towards Yb concentrations. ΔTC (ΔTC = TC onset – TC zero) had been broadened since the accretion Bi-2212 in each level of Yb concentration which attributed to the worse grain boundary properties and also modification of Bi-2212 phase caused by Yb content. The ΔTC increased with decreasing TC had been discussed earlier by Mangapathi *et al*., [22]. The suppression of TC is due to the less contact area between the Bi-2223 grains. The transition width showed a broader transition that indicates the presence of impurities and more than one phase present in the sample which implying increasing the weak links between superconducting grains. This result is in agreement with XRD result that is discussed earlier where there is more than one phase (Bi-2223 and Bi-2212 phase) existed in the sample. Similar behavior of increasing in ΔTc by substituted other element were reported earlier [23].

Table 3 The value of *Tc zero, Tc onset, ΔTc* and hole concentration for high-density and Low-density Yb-doped BSCCO

Yb concentration	High density Yb-Doped BSCCO				Low-density Yb-doped			
	$T_{C\ onset}$ (K)	$T_{C\ zero}$ (K)	ΔT_C (K)	Hole Concentration, ρ	$T_{C\ onset}$ (K)	$T_{C\ zero}$ (K)	ΔT_C (K)	Hole Concentration, ρ
x = 0.000	110	100	10	0.1600	109	96	13	0.121
x = 0.025	110	97	13	0.1409	100	85	15	0.108
x = 0.050	110	78	32	0.1084	84	73	11	0.096
x = 0.100	88	78	10	0.1084	83	69	14	0.093
x = 0.200	84	77	7	0.1072	76	60	16	0.086

The results of decreasing TC are due to the existence of the secondary phase formation as confirmed by XRD analysis. The suppression of TC could be related to a structural phase formation that is decomposing Bi-2223 phase and enhancing Bi-2212 phase besides the existence of weak links between the grains. The presence of a significant concentration of Yb atoms at the grain boundary regions would naturally transform the superconductor-insulator-superconductor (SIS) type configuration into superconductor-normal-superconductor (SNS) type. Similar result have been supported by others researchers [4,5,7,8,11,14,15]. The two-step transition is an indication of excessive weak links [14]. The decrease in TC onset is also related to intra-grain properties [15]. The substitution of Ca2+ by Yb3+ degrade the superconducting properties of Bi-2223 by providing additional electron leading to a decrease of TC and other superconducting properties [5].

The critical current density, JC for all samples taken at 60 K and measured in zero magnetic fields is displays in Table 4. It is found that the JC of the samples decreases with the increasing of Yb concentration. Low-density samples have higher JC compared to high-density samples due to a better grains connectivity that was improved during the sintering process. A porous structure produced by sucrose has a large surface area and, therefore, improve the grains connectivity [24].

Table 4 Variation of J_c at 60K in High and Low-Density Yb-Doped Bi-2223

Yb concentration	High Density		Low Density	
	J_c (A/cm^2) 60 K	Density (g/cm^3)	J_c (A/cm^2) 60 K	Density (g/cm^3)
x = 0.000	7.46	5.301	7.63	4.832
x = 0.025	5.81	5.298	5.28	4.911
x = 0.050	2.68	5.280	3.60	4.947
x = 0.100	2.12	5.285	2.96	4.733
x = 0.200	1.25	5.283	2.23	4.750

The decrease of Jc might be due to the increases amount of Bi-2212 phase at grain boundaries, which in turn decreases the intergranular Josephson coupling energy with a consequent decrease of pinning of intergranular vortices [25]. The existence of Bi-2212 phase in Bi(Pb)-2223 superconductor suppresses the current-carrying capability due the problem of weak link and the presence of impurity phase does not act as the flux pining center [23].

4.0 CONCLUSION

The high and low-density Yb-doped Bi-2223 superconductor have been prepared via COP and SSR. High-density samples have a higher volume percentage of 2223 phase and critical temperature, TC for the same concentration of Yb compared to low-density samples. However, the critical current density, JC in low-density samples is higher compared to high-density samples due to the large surface area in a porous structure made by sucrose. The large surface area favors improving the grains connectivity during the sintering process. Substitution of Yb in Bi-2223 decreased the volume percentage of 2223 phase, TC and JC towards higher concentration of Yb. The crystallographic structure is tetragonal in a low concentration of Yb but changes to orthorhombic at higher Yb-doped.

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