Characterization of Physical and Electrical Properties of Bi$_{1.8}$Pb$_{0.4}$Sr$_2$Ca$_{2-x}$M$_x$Cu$_3$O$_y$ (M = Na, Mg, Ce) Superconductor

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Abstract. Bi$_{1.8}$Pb$_{0.4}$Sr$_2$Ca$_{2-x}$M$_x$Cu$_3$O$_y$ (M = Mg, Na, Ce) Superconductor with x = 0.15 made through the solid method has been characterized by its physical and electrical properties. The purpose of this study was to determine the type of dopant in the superconductor Bi$_{1.8}$Pb$_{0.4}$Sr$_2$Ca$_{2-x}$M$_x$Cu$_3$O$_y$ which affected the best resulting morphology and critical temperature (Tc). Characterization of physical properties to determine morphology was done using Scanning Electron Microscope (SEM) JEOL-390, while the characterization of electrical properties to determine critical temperature (Tc) was carried out using Cryogenic Magnet Teslatron. The results showed that the type of dopant substituted on the superconductor Bi$_{1.8}$Pb$_{0.4}$Sr$_2$Ca$_{2-x}$M$_x$Cu$_3$O$_y$ greatly affected the morphology and critical temperature (Tc) produced. The best type of dopant that produces the optimum critical temperature is dopant Mg with T$_{\text{onset}}$ = 120.2 K and T$_c$ zero = 108.4 K, where the morphology formed looks homogeneous with low porosity.

1. Introduction
Superconductivity occurs when current flows in certain materials without energy dissipation. This phenomenon is characterized by perfect diamagnetic behavior and has zero resistance below critical temperature (Tc). Superconductors indicated for type I superconductors or also referred to as low temperature superconductors (LTS) and type II superconductors are also called high temperature superconductors (HTS)(1)(2).

Among high-temperature superconducting (HTS) materials, the Bi-Sr-Ca-Cu-O (BSCCO) system is considered to be the most attractive superconducting material because of its high superconducting Tc and critical current density Jc(3). BSCCO has three different phases based on the Cu-O field numbers (n = 1, 2, and 3), usually referred to as Bi-2201, Bi-2212, and Bi-2223 with critical temperatures (Tc) 20, 80, and 110 K in a row. The most promising phase for the application is Bi-2223 because of the highest critical temperature (Tc). However, it is difficult to get a single phase 2223 due to low stability(4).

The structural modification and superconducting properties of BSCCO are carried out through the process of using doping oxide or different elements (5)(6)(7). Pb doping use in BSCCO superconductor
synthesis due to the similarity of ion size and valence of Pb atoms, which results in the substitution of Bi atoms by Pb at the BiO double layer (8). Therefore, Pb can stabilize BSCCO-2223 superconductor and increase the critical temperature ($T_c$) and phase volume fraction 2223(9). The use of other dopants such as MgO, Na, and Ce has been carried out in the manufacture of bismuth (Bi) superconductors by several studies. The use of MgO dopants can increase the proportion of the Bi-2223 phase and increase flux pinning (10) and can increase the critical temperature (11), the use of Na dopants can increase the $J_c$ value (12), whereas dopant Ce produces an increase in current density ($J_c$) and a critical temperature increase ($T_c$) (13).

Solid method is a method that is widely used in the manufacture of superconductors, including bismuth (Bi)-based superconductors, because the process is faster in synthesizing superconducting materials, a relatively low cost, and can produce BSCCO-2223 with fairly good quality (14). Therefore, in this study the solid method was chosen in the manufacture of Bi$_{1.8}$Pb$_{0.4}$Sr$_2$Ca$_{2-x}$M$_x$Cu$_3$O$_y$ (M = Mg, Na, Ce) superconductors with $x = 0.15$. Furthermore, superconductors of Bi$_{1.8}$Pb$_{0.4}$Sr$_2$Ca$_{2-x}$M$_x$Cu$_3$O$_y$ (M = Mg, Na, Ce) superconductors were characterized by their physical and electrical properties. The purpose of this study was to determine the type of dopant in the Bi$_{1.8}$Pb$_{0.4}$Sr$_2$Ca$_{2-x}$M$_x$Cu$_3$O$_y$ (M = Mg, Na, Ce) superconductors which affected the best resulting morphology and critical temperature ($T_c$).

2. Method

The precursors used in this study were Bi$_2$O$_3$ (Bismuth (III) Oxide) PA 98%, SrCO$_3$ (Strontium Carbonate) PA 96%, CaCO$_3$ (Calcium Carbonate) PA 99%, CuO (Copper (II) Oxide) PA 99%, PbO$_2$ (Lead (IV) Oxide) PA 97%, MgO (Magnesium Oxide) PA 99%, Na$_2$CO$_3$ (Sodium Carbonate) PA 98%, and CeO$_2$ (cerium oxide) PA 99%. The method used in this study is the solid method.

The initial step of all precursors is first weighed according to stoichiometry with the formula Bi$_{1.8}$Pb$_{0.4}$Sr$_2$Ca$_{2-x}$M$_x$Cu$_3$O$_{y+3}$ (M = Mg, Na, Ce) with the composition = 0.15. Then, mixing and grinding with mortar for 3 hours. The sample is then heated at 300 °C for 6 hours. After the sample is heated then the sample is crushed again for 6 hours. The purpose of this grinding is to increase homogeneity in the sample. Then, the sample is calcined at a constant temperature of 820°C for 20 hours. The purpose of this calcination is to release gases in the form of carbonates or hydroxides, so as to produce materials in the form of high purity oxides. After calcination is carried out the cooling process in the furnace is closed. After the cooling process, the powder is pressed (p ~ 379 MPa) into a pellet with a diameter of 10 mm and a thickness of 3 mm.

After the sample has been pelletized, the sample is sintered inside the furnace at a constant temperature of 850 °C for 30 hours. The purpose of this sintering is to reduce the number and size of pores, trigger grain growth, increase in density and shrinkage.

After going through the sintering process, all pellets were characterized. Characterization was carried out using Scanning Electron Microscope (SEM) JEOL-390 to determine morphology (physical properties) and Cryogenic Magnet Teslatron to determine resistivity (electrical properties)Bi$_{1.8}$Pb$_{0.4}$Sr$_2$Ca$_{2-x}$M$_x$Cu$_3$O$_{y+3}$(M = Na, Mg and Ce) superconductor phase 2223 with $x$ = 0.15. Data obtained through further testing is analyzed.

3. Result and Discussions

A. Characterization Using SEM

Testing using SEM (Scanning Electron Microscope) was carried out to obtain a morphological picture of the sample. Analysis of the results to obtain morphological images and composition of the SEM used in technique (Energy Dispersive Spectroscopy). Each sample will be analyzed at 30X, 100X, 500X, 1000X, 3000X, and 5000X magnifications (15).

The characterization results using Scanning Electron Microscope (SEM) JEOL-390 with 3000X magnification are shown in Figure 1. The morphology of the sample B2 (0.15% at Mg) in Figure 1 shows that the crystals are in the form of pieces and look homogeneous. Crystals in the C2 sample (0.15% at Na) also appear to be shaped as chips, but when compared to the B2 sample, it looks more
homogeneous and tends to clot. While the morphology shown in the D2 sample (0.15% at Ce) crystals in the form of melted granules.

In addition to producing surface morphology images with magnification of 30X, 100X, 500X, 1000X, 3000X, and 5000X, SEM also produces mapping to determine the distribution (distribution) of elemental composition in a material. The result of Bi$_{1.6}$Pb$_{0.4}$Sr$_2$Ca$_{2-x}$M$_x$Cu$_3$O$_3$0.8 (M = Mg, Na, Ce) with x = 0.15% or B2 mapping is shown in Figure 2, which shows that the elements of Bi, Pb, and Sr are distributed with the highest concentration, Mg and O is distributed with the lowest concentration, while Ca and Cu are distributed with a higher concentration than Mg and O.

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\begin{align*}
\text{B2} & = 0.15 \% \text{at Mg} \\
\text{C2} & = 0.15 \% \text{at Na} \\
\text{D2} & = 0.15 \% \text{at Ce}
\end{align*}
\]
B. Characterization Using Cryogenic Magnet

Cryogenics is the study of the production and behavior of materials at very low temperatures. Cryogenic magnets are vacuum technology and gas compression/expansion (pulse tube probes) based on the Four Point Probe (FFP) principle useful in lowering Helium gas temperatures. Using the pulse tube probe method, the helium gas temperature drops by about 7K, then by the VTI method (the Helium gas pressure drop) the temperature drops to 1.5K, but the VTI pump’s cryogenic power decreases, so it can only lower the temperature to around 5K. This measurement method is one method for measuring resistivity or the method used to determine the Tc (critical temperature) of a material (16)(17)(15).

The results of characterization using cryogenic magnets to determine the resistivity (electrical properties) of superconductors Bi1.6Pb0.4Sr2Ca2-xMxCu3O10+δ (M = Mg, Na, Ce) are shown in Table 1 and Figure 3 below.

Table 1. Critical temperature testing (Tc) uses cryogenic magnets

| Sample Code | %at dopan | Tc onset (K) | Tc zero (K) |
|-------------|-----------|-------------|-------------|
| B2          | 0.15 Mg   | 120.2       | 108.4       |
| C2          | 0.15 Na   | 85          | 75.5        |
| D2          | 0.15 Ce   | 88.5        | 75.5        |

Table 1 shows that pending Mg on the superconductor Bi1.6Pb0.4Sr2Ca2-xMxCu3O10+δ with the composition of x = 0.15 results in the highest T onset and zero Tc compared to the Na and Ce doped. The Tc value is also higher when compared to previous studies (18) in the manufacture of BPSCCO-2223 superconductor with dopants Mg (x = 0.2) which produces Tc onset = 117.5 K and Tc zero = 101.7 K.
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