The Influence of MgO Addition on the Fabricating BPSCCO Superconducting Monofilament Wires using Ag-sheated and Stainless Steel 316 Tubes Prepared By PIT Method

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Abstract. The synthesis of MgO particles were substituted into Bi1.6Pb0.4Sr2Ca2-xOxCu3Oy superconducting monofilament wires using Silver and Stainless Steel 316 tubes by Powder-In-Tube method was done. Phase identification by X-Ray Diffraction (XRD), microstrcture analysis by SEM-EDS and the resistivity as a function of temperature measurement. The effects of addition 5% wt MgO in the sample of BPSCCO superconducting monofilament wires using Ag-sheated and Stainless Steel 316 on the critical temperature (Tc) of pure and MgO dopant were sheated by Ag tube with Ag-sheated with sintering time 9 h and 30 h with produced the Tc-onset 98 K; 78 K; 72 K. The sample was sheated Stainless Steel 316 produced the Tc-onset = 92.9 K for BPSCCO without dopant, however the critical temperature for BPSCCO with the addition of MgO dopant did not show. The surface morphology, is analyzed by SEM-EDS and the results shows that the crack and the grains distributed randomly. The diffraction pattern of the sample with XRD and the addition of MgO dopant to BPSCCO superconducting monofilament with Ag and Stainless Steel 316 decreases the Tc-onset if compared sample without dopant. This is consistent with the decrease in fraction volume-2223 fraction and the increase in fraction volume-2212.

1. Introduction
The potential for high temperature superconductors (HTS) has a major impact on power technology. This is because the superconductor cable can deliver large amounts of electricity effectively in the underground space. In addition, construction costs are smaller than conventional cables. In high capacity power transmission on high temperature superconductor cables (HTS) have the advantage of efficiency against the cheap and environmentally friendly media representation [1].

The structure of HTSs on CuO2 plane and insulation layers are periodically laminated along c-axis direction. Especially, the precursor of Bi-2223 has unique features that work excellently and c-axis oriented texture is compromised with the weak connected portions of Bi-O double layers [2].

Loroux et al., reported that the partial replacement of Bi of Pb and Sb atoms permit synthesis of stable combination superconducting properties of the combination and an increase in Tc [3]. Doping
Mg and Be at the Ca site can improve the properties of superconductors and diamagnetism. This is due to the greater electromagnetivation value and the smaller ion size Mg and Be as compared to Ca. This can be proved by interplanetary coupling is also improved and shrinking of the axis length [4].

The effect of MgO addition can increase the proportion of the Bi-2223 phase and increase the flux pinning [5]. The effect of MgO decreases the size of the Bi-2223 grains where MgO as a barrier, inhibits grain growth and affects the microstructure. In the process during sintering, grain boundary migration and element values differ. MgO influences the magnetic field hysteresis loop significantly so that the value of 𝐽c increases with the addition of MgO not more than 15% wt [6]. Stainless Steel 316 tube is a type of austenitic Stainless Steel. The austenitic alloy has not only excellent corrosion resistance properties, but also has additional properties such as easily shaped and fabricated [7].

The fabrication methods on the cable manufacturing process, wire-shaped bulk samples commonly use the Powder-In-Tube method for fabrication of wire. The Powder-In-Tube method is simple methods both in terms of mixing materials, the process of inserting powder into the tube and sintering process [8].

Because of its structure, the Bi-2223 material is suitable for the Powder-In-Tube (PIT) method. A unit cell of Bi-2223 has a lamination structure that contains a superconducting Cu-O layer and two insulating layers above and below the Cu-O layer. Moreover, Bi-2223 easily cleaves because of the relatively small bonding force between the double Bi-O layers within each insulating layer. It means in spite of their poor ductility, the filaments include the Bi-based superconductors can be drawn along with silver or silver alloy during the drawing step, and the oriented the c-axis direction during the rolling step. Therefore, the PIT method can be used in the manufacturing of Bi-2223 wires [2, 9].

2. Experimental Method

The precursor materials were prepared from a mixture of Bi₂O₃ (98%), PbO₂(97%), SrCO₃ (96%), CaCO₃ (98%) and CuO (99%) with PIT method. The main advantage of this method is the capability to generate a high critical current density (𝐽c) and to remove the defects caused by the casing [10]. The weight of the powder was measured using stoichiometry calculation (x = 0.00, 0.05), the monofilament billet of 8 mm diameter to 6 mm diameter was rolling and sintered in furnace at constant temperature 850°C for 𝑡₁ = 30 h and 𝑡₂ = 9 h. The equation of the BPSCCO-2223 with dopant MgO can be used with the following formulas:

\[
0.8\text{Bi}_2\text{O}_3+0.4\text{PbO}_2+2\text{SrCO}_3+1.95\text{CaCO}_3+0.05\text{MgO}+3\text{CuO} \rightarrow \text{Bi}_{10.6}\text{Pb}_{0.4}\text{Ca}_{1.95}\text{Mg}_{0.05}\text{Cu}_{10-3}\text{O}_{10-3}+4\text{CO}_2
\]  

Sintering time in furnace at constant temperature 850°C the formation of the Bi-2223 phase with the addition of MgO is also affected by the treatment of the sintering temperature used. At a temperature of 845°C with the addition of MgO will decrease the Bi-2223 phase shown in the inhibition of the rate of reaction due to the Bi-2223 formation. On the contrary, at sintering temperatures above 845°C, the addition of MgO influences the ratio of the Bi-2223 phase is not activated when the temperature increases. The more obvious elemental shapes and limiting the formation of the Bi-2223 does not have a significant impact on the amount of powder MgO [6, 11]. It is known that the sintering temperature increase is effective in controlling morphological grains and grains coupling to improve the properties of superconductors such as 𝑇c, 𝐽c and 𝐹p [12].

The characterization of the microstructure of the sample was observed with a Scanning Electron Microscope (SEM) JEOL-6390A and Energy-Dispersive X-ray Spectroscopy (EDS) for analyzes chemical elements of sample. The resistivity analysis of sample by the Teslatron Cryogen Free, Oxford Instrument system using helium gas as a working medium. The crystal structures were analyzed using XRD (X-Ray Diffraction, Rigaku Mini Flex 600).

3. Results and Discussion

First, we deal with microstructure morphology and the grain size of the sample BPSCCO/MgO superconducting monofilament wires using Silver and Stainless Steel 316 tubes with addition of 5 %wt
MgO. The Figure. 1 shows that the presence of crack caused by rolling process on wire, sample homogeneity has been quite good, although the resultant grain melts and each grain grows in random directions.

**Figure 1.** SEM images of BPSCCO superconducting monofilament dopant MgO (a) Ag-sheated with sintering time of 30 h, (b) Ag-sheated with sintering time of 9 h, (c) Stainless Steel 316 with sintering time of 30 h

**Figure 2.** Energy Dispersive X-ray Spectroscopy (EDS) spectrum of Sample BPSCCO superconducting monofilament dopant MgO (a) Ag-sheated (b) Stainless Steel 316-sheated
The sensitivity of SEM-EDS’ sample (a) shows that Bi element has the largest mass with 33.84% mass and 0.37% atom, the largest atom’s percentage presents in the element Cu with 21.11% atom and 19.07% mass, dopant MgO is 0.32% atom and 0.11% mass and mass’ percentage of Ag is 24.77% and 12.28% atom.

For sample (b) shows that the largest mass’ percentage in the Ag element is 25.51 and 0.26% atom, MgO element is 0.06% mass with atom’s percentage is 0.14 and Fe element mass’ percentage is 17.03 with 0.28% atom. shows that the addition of MgO element affects the shape of the crystals, the percentage of atoms, and the percentage of mass at BPSCCO superconducting monofilament dopant with Ag-sheathed and Stainless Steel 316.

The volume fraction value equation Bi-2223 and Bi-2212 on a sample can be used the following equation [13]:

\[
Bi-\text{(2223)%} = \frac{\Sigma I(2223)}{\Sigma I(2223)+I(2212)+I(\text{Impureties})} \times 100
\]

\[
Bi-\text{(2212)%} = \frac{\Sigma I(2212)}{\Sigma I(2223)+I(2212)+I(\text{Impureties})} \times 100
\]

where: I(2223) and I(2212) are the intensities of Bi-2223 and Bi-2212 phases respectively.

Table 1. Volume Fraction of Phase % and Critical Temperature for Bi\(_1.6\)Pb\(_0.4\)Sr\(_2\)Ca\(_2\)-\(\text{X}\)O\(_x\)Cu\(_3\)O\(_y\) Superconducting Monofilament Wires using Silver and Stainless Steel 316 Tubes (x = 0.00 ; 0.05) with sintering time 850 °C

| No | Sample       | Tube                | Sintering Times (hours) | Diameter (mm) | T\(_c\)-Onset (K) | T\(_c\)-zero (K) | Volume Fraction | Sample Code |
|----|--------------|---------------------|-------------------------|---------------|------------------|----------------|-----------------|-------------|
| 1. | BPSCCO       | Ag Stainless Steel 316 | 24                      | 5             | 98              | 72             | 26              | 74          | A           |
|    |              |                      | 24                      | 6             | 92.9            | 50.8           | 64.36           | 35.63       | B           |
| 2. | BPSCCO/MgO   | Ag Stainless Steel 316 | 9                       | 6             | 78              | 58             | 41              | 59          | A1          |
|    |              |                      | 30                      | 6             | 72              | 33             | 40              | 60          | A2          |
|    |              |                      | 30                      | 4             | -               | -              | -               | B1          |             |

Table 1. shows that the addition of MgO for BPSCCO superconducting monofilament wire using Ag-sheated and Stainless Steel 316 decreases T\(_c\)-onset if compared to the sample without dopant. This is consistent with the decrease in fraction volume-2223 and the increase in fraction volume-2212.

Bintoro. et al., reported the addition of MgO destabilizes the phase, increasing the Bi-2212 volume fraction and decreasing the Bi-2223 volume fraction. The resistivity curve shows a smooth curvature that is Bi-2223 phase one-step transition for pure and MgO addition These are the main causes of the decline in Tc-onset [14].

A metallic behavior a linear variation of resistivity with respect to temperature is observed in all samples at high temperatures (T >T\(_c\)), followed by a transition to zero resistance as temperature is reduced. T\(_c\)-onset can give information about the superconducting phases occurred in the grain structure, but T\(_c\)-offset generally gives information about weak links of the superconductor grains and grain boundaries [15].
In Figure 3. XRD patterns of BPSCCO superconducting monofilament wire using Ag and Stainless Steel 316-sheated

In Figure 3 shows a change of diffraction patterns of sample BPSCCO superconducting monofilament wires using Ag-sheated without dopant and BPSCCO with the addition of MgO had been changed. The samples show that the CuO impurity phase was formed in each sample with each intensity value of 990.32 cts; 993.77 cts; 902.86 cts. This indicates a critical has decreased temperature.

In the sample of BPSCCO superconducting monofilament wires using Stainless Steel 316-sheated without dopant and with the addition of MgO dopant undergo significant changes. It can be seen on the Stainless steel 316-sheated without using dopant has the formation of Bi-2223 and Bi-2212 phase at 2θ = 31.75 °, 2θ = 44.97. In the sample BPSCCO superconducting monofilament wires using Stainless Steel 316-sheated with MgO dopant formed Ni_{2.9}Cr_{0.7}Fe_{0.36}, Bi-2223 phase and Bi-2212 phase was not formed.
Figure 4. Resistivity as function of temperature of BPSCCO superconducting monofilament wires using Ag-sheated without dopant and with the addition of MgO

The resistivity as a function of temperature shows the comparison of BPSCCO superconducting monofilament wires using Ag-sheated with no dopant and dopant MgO with sintering time of 9 h. The result of identification of resistivity of sample A1 with sintering time 30 h and d = 5 mm. Shows that T_{c, onset} = 98 K and T_{c, zero} = 72 K. In addition of MgO dopant decreases T_{c, onset} = 78 K and T_{c, zero} = 58 K. This is due to the impurity phases in the sample. This can be seen with the decreasing Bi-2223 phase and the increase in the Bi-2212 phase seen in Table 1 [16][17].

For the sample of BPSCCO superconducting monofilament wires using Stainless Steel 316-sheated without dopant and with the addition of MgO shows that the one single step of transition to the superconducting state that’s in the pure sample and the addition of MgO does not indicate the presence of the transition temperature.

4. Conclusion
The results indicate that the effect of the addition 5% wt MgO in the BPSCCO superconducting monofilament wires using Ag and Stainless Steel 316-sheated on the phase formation, electrical properties and microstructure have been investigated. The critical superconductivity transition temperature (Tc) of sample without dopant and MgO dopant using Ag-sheated with sintering time 9 h and 30 h were found T_{c, onset} of 98 K; 78 K; 72 K. However, BPSCCO using Stainless Steel 316-sheated without dopant was produced 92.9 K, and the sample with addition of MgO did not show transition temperature.

The microstructure has been studied for the samples show that in the presence of crack caused by rolling process on wire, sample homogeneity has been quite good, although the resultant grain melts and each grain grows in random directions.

The phase formation with XRD analysis showed that MgO addition retards the reaction rate and hence the decrease of the volume fraction Bi-2223 and increase Bi-2212 phase.
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