The Effect of Sintering Time on Superconductor Wire Bi-Pb-Sr-Ca-Cu-O with Dopant Te Sheeted Ag Using Powder In-Tube Method

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Abstract. The use of silver and tellurium metals in fabricating superconductor material has been considered as promising materials. These metals are able to improve both mechanical and thermal stability. However, the fabrication of superconducting material is not merely affected by the composition of metals, but also the method in fabricating the material. To understand the effect of both factors, a study is conducted to investigate the effect of sintering time and dopant tellurium covered by silver sheet. The superconducting material was fabricated by powder in tube method composed with bismuth, lead, calcium, gold, and oxygen. The results showed that high time of sintering decreased the critical temperature of the superconducting material for over 20% from 65K to 51K. Morphological display showed that during high time of sintering caused melting of the grain even though the porosity was less. On the other hand, XRD pattern displayed that high time of sintering formed CuO and CaTeO₄ compounds considered as impurities. Overall, the length of time affects the superconducting properties including critical temperature and the purity of material.

Keywords: Sintering Time, Superconductor Wire, Dopant Te, Powder In-Tube Method, Sheeted Ag

1. Introduction

Superconducting BiₓSr₁₋ₓCaₓCu₂O₄₋ₓ+y in the compound formula with x = 1, 2, 3 is called the BSCCO phase 2201 (Tc= 20 K), BSCCO phase 2212 (Tc= 80 K) and BSCCO phase 2223 (Tc= 110 K). The bismuth-based superconductor is considered the most promising material, one of the most promising superconducting applications in the energy field of superconducting wire where its use in power distribution networks [1]. The problem found in these bismuth-based superconductors is the effort to increase the critical temperature and minimize the fragility of the material. Therefore, attempts were made to increase the critical temperature through various temperature variations at the time of the formation of superconductors and the variation of the cooling process of superconducting material when inside the heating furnace [2].

The Ag shell is usually mixed with one or more other metals to improve the mechanical properties of the wire and to obtain the characterization properties of the high temperature superconductors in it [3][4]. The stability of the structure is pursued by doping the superconducting material with the substitution of Pb material [5], [6]. Doping Pb can increase the critical temperature of BSCCO superconductors respectively by 106.42 K and 107 K [7][8], because it has physical and chemical properties similar to the Bi element so it can substitute the Bi position on BSCCO crystal system. Te doping ions can increase the mobility of some of the oxygen vacuum around them that make some characteristic changes at low temperatures [9]. In the double doping method Pb and Te substituting Bi can suppress the crystal growth of the whisker BPSCCO.

Doping of both Pb and Te may act as a catalyst to promote BSCCO phase decomposition or establish isolation contacts [10]. The optimum sintering temperature is about 845oC, which is the highest density value and the highest volume fraction of Bi-2223 based on XRD results. In addition, the XRD results show that with increasing sintering temperatures up to 845oC, the Bi-2212 phase fraction decreases. The SEM results show that the surface morphology of the sample is increased by increasing the sintering temperature to 845 °C. It is known that sintering temperature increase is effective in controlling morphological grains and grains coupling to the properties of superconductors such as Tc, Jc and Fp [1]. In the research of Hamadneh [11], the volume fraction of Bi (Pb) -2223 with sintering temperature of 850 °C and variation of time 24, 48, and 100
hours was 97; 97.5 and 98.7% respectively. The Powder-in-Tube (PIT) method is used in the manufacturing of Bi-2223 wire because it has a laminate structure containing superconductors.

2. Methodology

2.1 Materials.
The materials used in the synthesis of superconducting monofilament wire are Bismuth (III) Oxide, Lead (IV) Oxide, Strontium Carbonate, Calcium Carbonate, Copper (II) Oxide and 2% Tellurium dopant which is crushed for 3 hours with mortar. The initial mono-filament billet with a diameter of 8 mm is then rolled up to 5 mm in diameter.

2.2 Methodology
In this research, PIT method is commonly used for cable and cassette fabrication. This method involves a simple procedure of mixing all the starting materials, packing the powder in a metal tube, and sintering. The main advantage of this method is that it is capable of carrying high critical current densities and capable of removing defects caused by the cloak [12]. The recrystallization process subsequently sintered in the furnace at a constant temperature of 850°C for 30 and 9 hours then cooled in the furnace.

Characterisations
The test of correction factor of resistivity measurement (ρ) was conducted with four-point probe method using Cryogenic (Cryotron FR Oxford), characterization of structural properties using X-Ray Diffraction (XRD Rigaku Mini Flex 600), and to investigate surface microstructure of sample using Scanning Electron Microstructure (SEM JEOL type JSM6390A).

3. Results And Discussion

3.1 Difraction Pattern of Superconductor

Figure 1 (a) was a sample of BPSCCO Dopant Te superconducting monophyletic waves with a 30-hour sintering treatment, the figure showing that the formation of phases (Bi, Pb) -223, (Bi, Pb) -2212, CuO and CaTeO4. Phase (Bi, Pb) -2212 optimum at 2θ = 43.39° with intensity 20 cts, phase (Bi, Pb) -2223 optimum at 2θ = 38,74° with intensity 19 cts, phase envelope Ag is at 2θ = 38 , 24° with intensity 13 cts; 2θ = 44,44° with intensity 20 cts; 2θ = 64.70° with intensity 19 cts and 2θ = 77.68° with intensity 94 cts. CuO impurity phase is at 3 angle that is 2θ = 65.71° with intensity 19 cts; 2θ = 68.00° with intensity 6 cts and 2θ = 72.30° with intensity 9 cts, and CaTeO4 impurity phase is at 2θ = 22.67° with intensity 16 cts; 2θ = 43.59° with intensity 16 cts and 2θ = 37,78° with intensity 10 cts. The results show that BPSCCO with Te dopant improves the appearance of other impurity phases of CuO and CaTeO4 resulting in a critical temperature drop (Tc).

Figure 1 Diffraction Pattern of Monofilament Superconducting Wire Samples a. BPSCCO Dopant Te sintering 30 hours b. BPSCCO Dopant Te sintering 9 hours
Figure 1 (b) shows a sample of monofilament BPSCCO dopant Te superconducting waves with a 9-hour sintering, the figure shows optimum Phase (Bi, Pb) -2212 at $\theta = 29.09^\circ$ with intensity 9 cts, phase (Bi, Pb) -2223 optimum at $\theta = 56.50^\circ$ with intensity 5 cts, phase of Ag shell at $\theta = 38.26^\circ$ with intensity 7 cts; $\theta = 44.46^\circ$ with intensity of 6 cts; $\theta = 64.70^\circ$ with intensity 8 cts and $\theta = 77.68^\circ$ with intensity 21 cts; CuO impurity phase at 3 angle that is $\theta = 38.74^\circ$ with intensity 28 cts; $\theta = 35.56^\circ$ with intensity 6 cts and $\theta = 66.50^\circ$ with intensity 9 cts; CaTeO4 impurity phase at 4 angle that is $\theta = 18.24^\circ$ with intensity 6 cts; $\theta = 39.40^\circ$ with intensity 15 cts; $\theta = 40.72^\circ$ with intensity 5 cts and $\theta = 48.55^\circ$ with intensity 7 cts. Orthorhombic sample crystal structure with lattice parameter a = 5.347 Å, b = 5.416 Å and c = 30.67 Å.

From the analysis, it can be seen that the BPSCCO samples obtained had impurities which were CuO and CaTeO4. These impurities were presented as the Bi$_2$CuO$_4$ was decomposed when the temperature reached over 750°C. As a result, the critical temperature was affected so that it decreased.

The percentage of outcome volume fraction on monofilaments BPSCCO wire superconductor sample with dopant Te with 9-hour sintering treatment showed that the largest fraction of volume in the Bi-2212 phase was 64% while in the Bi-2223 phase was 36%.

Figure 2 shows the results of the resistance test of monofilament wire BPSCCO dopant Te wire with Agent cylinder sheath using Cryogenic. The images are the result of resistance identification from BPSCCO dopant Te diameter 5 mm sample with 30 hour sintering time only show Te onset = 51 K, while in sample with time sintering 9 hours show Te onset = 84 K and Te zero = 65 K.
Figure 3 Sample Morphology Wire Superconductor BPSCCO Dopant Te Magnification 1000 x with a sintering time of 30 hours diameter 5 mm (a) appears to edge (b) centred

![Sample Morphology Wire Superconductor BPSCCO Dopant Te Magnification 1000 x with a sintering time of 30 hours diameter 5 mm](image)

Figure 4 Morphology of Wire Superconducting Samples BPSCCO Dopant Te Magnification of 1000 x with a sintering time of 9 hours diameter 5 mm (a) edge view (b) centred

![Morphology of Wire Superconducting Samples BPSCCO Dopant Te Magnification of 1000 x with a sintering time of 9 hours diameter 5 mm](image)

Figure 3 and 4 shows the morphological comparison of BPSCCO monofilament wire surface in diameter 5 mm with sintering temperature of 850°C for 30 hours and 9 hours. Figure 3 shows less porosity than in figure 4 due to longer heating time so that all grains melt evenly. The uniform grain melting causes the grains to become denser and look uniform which creates low porosity. In Figure 4, the porosities on the samples’ surface is bigger than those in figure 4 because of the shorter time of sintering.

**CONCLUSION**

The addition of sintering time on the monofilament wire sample of BPSCCO dopant Te 2% monofilament resulted in a decrease in the critical temperature. Sintering treatment with time 9 hours Tc onset = 84 K and Tc zero = 65 K while in sintering treatment with time 30 hours only show Tc onset 51 K. Based on the characterisation results, the samples obtained in the optimum critical temperature is the sample which had 9 hours of sintering. Dopant Te resulted impurities phases which were CuO and CaTeO₄, forming Bi, Pb–2212 phase.
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