Fabrication of SS316-Sheathed BPSCCO Superconducting Wire with the Addition of Carbon Nanotubes

Hilda Ayu Marlina¹, Agung Imaduddin², Kerista Sebayang¹*, Pius Sebleku³, Satrio Herbirowo², Hendrik²

¹Department of Physics, University of Sumatera Utara
Jl. Bioteknologi No. 1, Medan 20155, Sumatera Utara, Indonesia
²Research Center for Metallurgy and Materials – Indonesian Institute of Sciences (LIPI) Gedung 470 Kawasan PUSPIPTEK, Tangerang Selatan, Indonesia

*E-mail : keristasebayang@usu.ac.id

Abstract. This study aimed to determine the effects of using SS316 as a sheath of BPSCCO superconducting wire with the addition of 5 wt% carbon nanotubes. The method used in this research was powder in the tube (PIT), which consisted of several steps; namely preparation of precursors (Bi₂O₃, SrCO₃, CaCO₃, CuO₂, PbO₂, and carbon nanotube powder); manual mixing precursors using mortar agate for 3 hours; compaction of precursors in tubes; rolling the wire with the diameter up to 5 mm; calcinations at 820 °C for 20 hours; and sintering at 850 °C with time variation of 30 and 9 hours. The characterization was performed using cryogenic magnet to determine the value of critical temperature (Tc), SEM-EDX to analyze morphology, and XRD to discover the phase formed. From the research, it was found that the use of SS316 as a sheath for BPSCCO superconducting wire with the addition of 5 wt% carbon nanotubes affected the Tc. The system only produced the Tc onset of 100 K for the sample sintered for 30 hours, whereas the Tc zero was not produced.

1. Introduction
Superconducting material was first discovered by Onnes in the early 1900s. The superconducting material loses its electrical resistance and rejects the magnetic field when cooled under the critical temperature called Tc (1). In 1989, high-temperature superconductors (HTS) or also known as copper oxide were found. HTS are in great demand for scientists to develop. In 1994, several American departments, such as the Department of Energy (DOE) and the Department of Defense (DOD), have sponsored the development of HTS. The program aimed to commercially develop the HTS applications as a more efficient and flexible energy system (2)(3).

The toxicity present in some HTS superconductors such as thallium-based superconductors (TBCCO) and mercury-based superconductors (HBCCO) make it possible for Bismuth-based superconductors (BSCCO) to be studied (3). In BSCCO system, there are 3 superconducting phases which have been identified, those are Bi-2201 phase with stoichiometric composition Bi₂Sr₂CuO₆+δ and Tc about 24 K; phase Bi-2212 with stoichiometric composition Bi₂Sr₂Ca₂Cu₃O₆+δ and Tc between 80-96 K; and Bi-2223 phase with stoichiometric composition Bi₂Sr₂Ca₂Cu₃O₁₀+δ and Tc 110 K (4). The Bi-2223 phase superconductor attracts many researchers’ attention because it has a high Tc value. Moreover, the enhancement of the superconducting properties of this material has been carried out
with a different approach. One of them is based on structural modification and superconducting properties through the process of using different doping oxides or metal elements (5) (6). The use of Pb doping in BSCCO superconductor synthesis is 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. Therefore, the substitution of Pb can stabilize the BSCCO-2223 superconductor and increase the critical temperature (Tc) and phase volume fraction 2223 (7). In addition, the use of doping carbon nanotubes has also increased the cuprate properties (Dadras, Liu, Chai, Daadmehr, & Kim 2009) (9)(10)(11), because it is known to reduce grain size so that it can increase the critical current density (Jc) (12). In addition, the carbon nanotubes vacuum chamber itself has a high current density, even higher than metals and superconductors (13), which ranges from $10^9$-$10^{10}$ Ampere/cm$^2$ (14)(15)(16).

Applications of the HTS development include mass and microelectronic devices, such as motors, generators, transformers, power transmission cables, energy storage systems, radio frequency (rf), microwave passive components, radar subsystems, wireless communications and medical instrumentation (2) (17). To achieve the success of these superconductor applications, the fabrication of superconducting wire is developed. The method of powder in tube (PIT) is one of the techniques in the fabrication of HTS superconducting wire. This method was first developed by the company Vacuumschmelze in Germany and Sumitomo Electric Industry (SEI) in Japan (3)(18). The PIT method consists of mixing precursors, compressor precursors in tubes, calcination, wire rolling, and sintering (19)(20).

The Ag tube is often used as a sheath in the fabrication of HTS superconducting wires, but, the price of Ag tube is quite expensive when compared with stainless steels type 316 (SS316) tube, which is around 1:96. In a previous study, it was stated that the use of SS316 tube in the fabrication of BPSCCO superconducting wires resulted in Tc onset value of 92.9 K and Tc zero value of 50.8 K (21).

In this research, BPSCCO superconducting wire was made by adding 5% wt carbon nanotubes covered by SS316. This study aims to determine the effects of using SS316 as a sheath of BPSCCO superconducting wire with the addition of 5 %wt carbon nanotubes, it is hoped that it can replace Ag’s use because it is expensive.

2. Method
The materials used are Bismuth (II) Oxide (Bi$_2$O$_3$) powder with purity of 98% PA, Strontium Carbonate (SrCO$_3$) powder with purity of PA 96%, Calcium Carbonate (CaCO$_3$) powder with purity of PA 99%, Copper Oxide (CuO$_2$) powder with purity PA 99 %, Lead Oxide (PbO$_2$) powder with purity of PA 97%, carbon nanotubes powder with purity of PA> 99%, and SS316 tube.

The tools used in the fabrication of BPSCCO superconducting wire with addition of 5 %wt carbon nanotubes sheathed by SS316 are digital scales, crusher (pastel and Agate mortar), heating furnaces, pellet molds, crucible, press machines, rolling tools. While the equipments for testing in this study are Cryogenic Magnet, Teslatron Oxford Instrument type, XRD type Rigaku Miniflex 600, and SEM EDS brand Zeiss & Jeol.

The first stage of the fabrication BPSCCO superconducting wire with addition of carbon nanotubes sheated by SS316 was preparation and weighing of Bi$_2$O$_3$, SrCO$_3$, CaCO$_3$, CuO$_2$, and PbO$_2$ with stoichiometric calculation of BPSCCO phase 2223. Then, carbon nanotubes powder was added 5 %wt from mass of BPSCCO powder phase 2223. Mixing of precursors was performed using mortar for 3 hours.

Before the powder is inserted into the SS316 tube, solutionize the SS316 tube to remove impurities. Cooling is done using quenching method at 900 °C for 1 hour. Quenching is done to process the strain hardening tube SS316 used. After quenching the tube is done, the SS316 tube is cleaned using sandpaper paper, then, cleaned again using 1 molar of HCl.

BPSCCO powder with the addition of 5 %wt of carbon nanotubes calcined at 820 °C for 20 hours. Then, the powder is inserted into SS316 tube which has diameter of 8 mm and a length of 5 cm. Emphasis was done manually. BPSCCO superconducting wire with the addition of 5 %wt carbon
nanotubes sintered at 850 °C for 30 hours. Then, the wire rolling with an initial diameter of 8 mm to 4 mm. Next, step was cutting of the wire into two parts, the first part was sintered at 850 °C for 30 hours and the second part was sintered at 850 °C for 9 hours.

The BPSCCO superconducting wire with the addition of 5 %wt carbon nanotubes sheathed by SS316 was characterized using cryogenic magnets to determine the Tc (critical temperature) obtained, SEM to analyze the morphology, and XRD to analyze the formed phases. XRD data measurements were analyzed using Match v1.10 software in accordance with the 2003 International Center for Diffraction Data (ICDD) database.

3. Result and Discussions

The results of critical temperature testing (Tc) using cryogenic magnet in the fabrication of BPSCCO superconducting wire with the addition of 5 %wt carbon nanotubes sheathed by SS316 are shown in Figures 1(a) and 1(b) as follows.

Figure 1. Relation of Resistance to Temperature: (a) S1 Sample and (b) S2 Sample

Figure 1(a) shows that the superconducting wire characteristic graph of S1 sample is not formed, because from the Tc test (critical temperature) using cryogenic magnet only Tc onset produced of 100 K, but Tc zero is not obtained. While on S2 sample, Tc onset and Tc zero, both are not formed. This can be seen in Figure 1(b) which shows that the graph of the resistance relationship to the temperature generated in the sample S2 is indicated as a graph of resistance to temperature characteristics of the conductor material.

In the previous study, on the fabrication of BPSCCO superconducting wire without dopants using SS316 tubes produced Tc onset of 92.9 K and Tc zero of 50.8 K of 6 mm diameter wire (21). Compared to this research the actual Tc onset obtained was larger; only Tc zero was not obtained.

The result of Tc (critical temperature) test obtained for BPSCCO superconducting wire with addition 5 %wt carbon nanotubes sheathed by SS316 is summarized in Table 1 below.
The XRD test results for S1 samples are shown in Figure 2 as follows.

![Figure 2. Diffraction Pattern of S1 Sample](image)

From Figure 2 it can be seen that the phase formed in the S1 sample is the phase $\text{Bi}_{0.96}\text{Pb}_{0.24}\text{SrCaCu}_{1.6}\text{O}_{5+x}$ with CuO and CaCO$_3$ impurities, while the Bi(Pb)-2212 and Bi(Pb)-2223 phases are not formed. When compared with the S2 sample as a whole, this S1 sample is the best sample because it produces 100 K of Tc onset even though Tc zero is not formed.

![Figure 3. The morphology of (a) S1 Sample and (b) S2 Sample Magnification of 5000x appears to be Middle](image)
The morphological of S2 sample in Figure 3(a) shows a more homogeneous crystals of the S1 sample, but appears to agglomerate. However, the porosity in the sample S2 appears to be larger than the S1 sample, possibly causing the critical temperature (Tc) of the superconductor not formed.

Some previous studies showed that carbon nanotubes and stainless steels have good adhesion properties (22). Another study showed that the catalytic growth of carbon nanotubes in stainless steel was very good (Abad et al., 2008) (23). Based these studies, it can be indicated that the interaction of carbon nanotubes with stainless steels during the sintering process causes the difficulty of BPSCCO superconducting phase formation in BPSCCO superconducting wire with the addition of 5 %wt carbon nanotubes sheathed by SS316. This causes the only formation of onset in the S1 sample and in the sample S2 both Tc onset and Tc zero are not formed.

4. Conclusions
Based on the results obtained in the study, it was concluded that the fabrication of BPSCCO superconducting wire with addition of 5 %wt carbon nanotubes sheathed by SS316 affects the Tc (critical temperature) formed. The optimal of Tc produced in the S1 sample, ie the sample was sintered at 850 °C for 30 hours, with Tc value of 100 K, even though Tc zero is not formed. The Tc zero was not formed because of indicated by the interaction between carbon nanotubes and SS316 during the sintering process.

5. Acknowledgment
The authors are grateful to LIPI 2017 Superior Fund and LIPI Research Center of Metallurgy and Materials of South Tangerang, Indonesia.

References
[1] Abbas MM, Abbas LK, Bahedh H. Superconducting Properties of Bi2-xSbxPb0.3Sr0.1Ca2Cu3O10+δ Compounds. J Appl Sci Res. 2015;11(22):164–72.
[2] Lubkin GB. Power Applications of High-Temperature Superconductors. Phys Today. 1996;49(3):48–51.
[3] Balachandran U, Iyer AN. Status of High-Tc Superconductors. Mater Technol. 1996;11(4):145–52.
[4] Nilsson A. BSCCO Superconductors Processed by The Glass-Ceramic Route. Arbeit. 2009.
[5] Ilyushechkin AY, Agranovski IE, Altman IS, Choi M. Effect of MgO nanoparticles embedded into Bi-2212/Ag tapes on the microstructure and superconducting properties. Mater Sci Eng B Solid-State Mater Adv Technol. 2010;167(1):60–4.
[6] Özkurt B, Madre MA, Sotelo A, Diez JC. Structural, superconducting and mechanical properties of molybdenum substituted Bi1.8Sr2Ca1.1Cu2.1Oy. J Mater Sci Mater Electron. 2013;24(4):1158–67.
[7] Hooker MW. Preparation and Properties of High-Tc Bi-Pb-Sr-Ca-Cu-O Thick Film Superconductors on YSZ Substrates. 1996.
[8] Dadras S, Liu Y, Chai YS, Daadmehr V, Kim KH. Increase of Critical Current Density with Doping Carbon Nano-tubes in YBa2Cu3O7-δ Compounds. J Phys Condens Matter. 2009;21:136469. Available from: http://dx.doi.org/10.1088/0953-8984/21/13/136469.
[9] Galvan DH, Durán A, Castillón FF, Adem E, Escudero R, Ferrer D, et al. Enhancement of The Current Density Jc for Bi2Sr2CaCu2O8 by Means of Carbon and NbSe2 Nanotubes. J Supercond Nov Magn. 2008;21:271–7.
[10] Galvan DH, Li S, Yuhasz WM, Kim JH, Maple MB, Adem E. Nondestructive interactions of carbon nanotubes with Bi2Sr2CaCu2O8 Phys C [Internet]. 2004;403:145–50. Available from: http://linkinghub.elsevier.com/retrieve/pii/S0921453403017052.
[11] Yang ZQ, Su XD, Qiao GW, Guo YC, Dou SX, Boer FR de. Flux-pinning Enhancement in Ag-sheathed Bi-2223 Tapes by Nanometer-SiC Addition. Phys C. 1999;325:136–42.
[12] Galvan DH, Kim J, Maple MB, Hirata GA, Adem E. Flux Pinning Effect of Embedded Carbon Nanotubes in Bi2Sr2CaCu2O8. Phys C. 2000;341–348:1269–70.
[13] Lekawa-Raus A, Gizewski T, Patmore J, Kurzepa L, Koziol KK. Electrical transport in carbon nanotube fibres. Scr Mater. 2017;131:112–8.
[14] Wei BQ, Vajtai R, Ajayan PM. Reliability and Current Carrying Capacity of Carbon Nanotubes. Appl Phys Lett. 2001;79(8):1172–4.
[15] Dew-Hughes D. The Critical Current of Superconductors: An Historical Review. Low Temp Phys. 2001;27(9–10):967–979.
[16] Yao Z, Kane CL, Dekker C. High-Field Electrical Transport in Single-Wall Carbon Nanotubes. Phys Rev Lett. 2000;84(13):2941–4.
[17] Lubkin GB. Applications of High-Temperature Superconductors Approach the Marketplace. Phys Today. 1995;48(3):20–3.
[18] Sandhage K, Riley G, Carter W. Critical Issues in the Opti Processing of High-Jc BSCCO Superconductors. Jom-Journal Miner Met Mater Soc [Internet]. 1991;43(3):21–5. Available from: https://apps.webofknowledge.com/full_record.do?product=UA&search_mode=GeneralSearch&qid=1&SID=R1F7klaIDZzLmgVAmeQ&page=1&doc=5&cacheurlFromRightClick=no
[19] Rey CM, Malozemoff AP. Fundamentals of superconductivity. In: Superconductors in the Power Grid [Internet]. Elsevier Ltd; 2015. p. 29–73. Available from: http://dx.doi.org/10.1016/B978-1-78242-029-3.00002-9
[20] Malozemoff AP. The Power Grid and The Impact of High-Temperature Superconductor Technology: An overview. In: Superconductors in The Power Grid: Materials and Applications. 2015. p. 1–28.
[21] Lubis H, Imaduddin A, Sebleku P, Herbirowo S, Marlina H. The Influence of MgO Addition on the Fabricating BPSCCO Superconducting Monofilament Wires using Ag-sheated and Stainless Steel 316 Tubes Prepared By PIT Method. 2018.
[22] Li D, Cheng Y, Wang Y, Zhang H, Dong C, Li D. Improved field emission properties of carbon nanotubes grown on stainless steel substrate and its application in ionization gauge. Appl Surf Sci. 2016;365:10–8.
[23] Abad MD, Sánchez-López JC, Berenguer-Murcia A, Golovko VB, Cantoro M, Wheatley AEH, et al. Catalytic growth of carbon nanotubes on stainless steel: Characterization and frictional properties. Diam Relat Mater. 2008;17(11):1853–7.