

Monofilament Wire Silver Sheathed Bi,Pb-Sr-Ca-Cu-O Prepared by Four-Pass Rolling and Repeated Heating Process

Hendrik¹, P. Sebleku¹, S. Herbirowo¹, S. D. Yudanto¹, B. Siswayanti¹, Lusiana¹, H. Nugraha¹, A. Imaduddin¹ and A. W. Pramono¹

¹Research Center for Metallurgy and Materials-Indonesian Institute of Sciences (P2MM-LIPI) Kawasan Puspiptek, Serpong, Tangerang 15314, Indonesia.

E-mail: hend027@lipi.go.id

Abstract. A four-step process was proposed to manufacture monofilament wire Bi,Pb-Sr-Ca-Cu-O using silver (Ag) sheath fabricated by Powder-In-Tube (PIT) method in which the thickness of wire were subjected to rolling from 10 mm Ø to 2 mm Ø and heated at temperature of 860 °C in 24 h. The effect of the rolling and heating process on the properties of four samples Ag-sheathed Bi,Pb-Sr-Ca-Cu-O wires in each step were studied. In x-ray diffraction (XRD) patterns of monofilament wire fabricated by PIT method in which the repeated heating process involved in rolling can alter the precursor phase. At a first and second deformation process, samples sintered at 860 °C in 24 h still contained the constituent elements of precursor powders. Third deformation step were shown to have superconducting phase Bi,Pb-Sr-Ca-Cu-O 2212. According to resistivity measurement analysis, rolling and repeated heating process were shown to have a critical effect on superconductivity. First, second and fourth rolling were found to have no superconductivity, while third rolling were found to exhibit superconductivity of which its value of critical temperature monofilament wire ($T_{c,onset}$) was 90 K. Furthermore, according to Scanning Electron Microscopy (SEM) analysis, the rolling and repeated heating process were observed to have important effect on morphology of samples. Wire superconductors on first and second rolling were shown to have crack and high porosity, but after third rolling the crack and porosity were diminished. Overall the deformation process have yielded promising results particularly when the superconducting phase Bi,Pb-Sr-Ca-Cu-O 2212 were found optimum in third deformation process.

Keywords. Monofilament and PIT method.

1. Introduction

Throughout the ten decades, the technological applications of superconductivity such as magnet and electrical power application have been used for long-distance and low-voltage electric grids without transmission loss; fast magnetically levitated trains; superefficient motors and generators where some of them are in demonstration and experimental stage. Therefore, many researchers have devoted their efforts on composites of superconducting materials and metals especially they own better mechanical properties and electrical properties than superconducting materials alone give [1–4]. As one of the promising high temperature superconductors, Bi,Pb-Sr-Ca-Cu-O (BPSCCO) have remarkable properties in the elongated forms such as wire and tapes for higher capacity superconductors cables of which some of them have been produced commercially by several companies around the world [5].



However, other superconducting materials such as low temperature superconductors (NbTi, Nb₃Sn, Nb₃Al) is also attractive for this application.

On the one side, as composites of superconducting materials and metals are brittle and difficult to fabricate, many researchers reported how they formulated these composites of superconducting materials and metals into three stages consisting of the precursor powder preparation stage [6–7], composites precursor preparation stage [8] and thermomechanical processing stage [9]. Among these composites, cuprate superconductors is believed to be the best potential candidate for a large scale of the technological application of superconductivity since it is operating in above the boiling point of liquid nitrogen range, of which the use of liquid nitrogen is environmentally clean coolants instead of oil. On the other side, previous researchers reported that bubbles were serious problem during thermomechanical process [10].

In this research, the preparation of thermomechanical processing stage of the composite of BPSCCO and silver(Ag) sheath fabricated by PIT have been modified with four-pass rolling and repeated heating process. It is interesting that four-step deformation process influence the variation of $T_{c,onset}$ value and $T_{c,final}$ value. We observed the superconducting phase BPSCCO 2212 were found optimum in third deformation process. For this purpose, the addition of four-pass rolling and repeated heating process on the quality of superconducting phases was systematically investigated. The quality of superconducting phases was analyzed from the particle size, crystallite structure and impurities.

2. Materials and methods

The preparation of composite of Bi,Pb-Sr-Ca-Cu-O (BPSCCO) using silver (Ag) tube fabricated by traditional PIT (Powder-In-Tube) method through the rolling and repeated heating process were attempted. In Figure 1, the flowchart described a four-step process of making monofilament wire BPSCCO. Based on the stoichiometry in first step, in order to obtain the phase of Bi_{1.6}Pb_{0.4}Sr₂Ca₂Cu₃O_{6+α}, the weight ratio of powders Bi₂O₃, PbO, SrCO₃, CaCO₃, CuO was fixed at 4.17:1:3.3:2.24:1.78. In second step, powders were mixed and inserted into Ag tube with initial thickness of Ø 10 mm. In third step, the Ag tube was rolled into diameter of Ø 8 mm to avoid crack. In fourth step, the composite of BPSCCO and Ag tube were heated in an electric furnace at 860 °C for 24 h under atmospheric air atmosphere. The thickness reductions of all samples during four-pass rolling process were 8 mm, 6 mm, 4 mm and 2 mm, respectively, as illustrated in Table 1.

Table 1. Process of making monofilament BPSCCO with four-step process

Step	Rolling	Heating
1	10 mm to 8 mm Ø	860 °C, 24 h
2	8 mm to 6 mm Ø	860 °C, 24 h
3	6 mm to 4 mm Ø	860 °C, 24 h
4	4 mm to 2 mm Ø	860 °C, 24 h

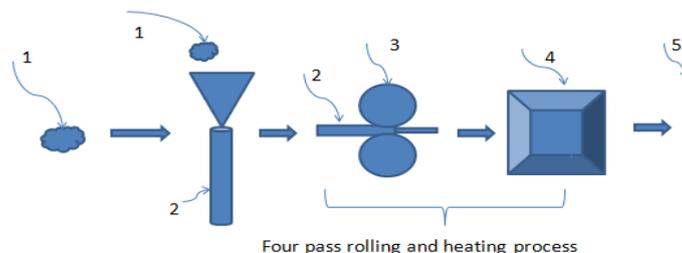


Figure 1. Flow chart of four-step process of making monofilament wire (Bi,Pb)SCCO In order to verify the quality of composite of superconducting BPSCCO and Ag, sample in each pass rolling process were investigated by four-point probe method, X-ray diffraction analysis and scanning electron microscopy (SEM) with an energy dispersive analysis (EDS).

3. Results and discussion

In Figure 2, the XRD patterns of monofilament wire BPSCCO 2212 using Ag sheath were obtained after peeling of the Ag sheath of sample sintering in air at 860 °C for 24 h for third pass rolling process. This is in agreement with the previous report [11]. As it can be seen in Figure 2a and Figure 2b, before third pass rolling, small peaks corresponding with Bi₂O₃ or BPSCCO constituent oxide powders are indicated. On the contrary, as indicated in Figure 2c, small peaks are observed corresponding to BPSCCO 2212, then disappears after fourth pass rolling because it is too small to be indicated.

In Figure 3, from the *R-T* curves showed the resistivity analysis of sample monofilament wire after third pass rolling. The present of the superconducting properties for sample after third pass rolling is quite interesting. The sample simulates the growing of BPSCCO 2212 with the superconducting state transition beginning at 90 K and completing at 70K, as is shown in Figure 2. This result points out the activation for the reaction between powders due to heating after 48 h.

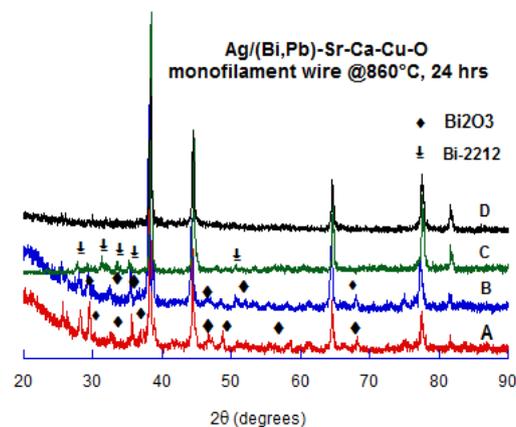


Figure 2. X-ray diffraction patterns of monofilament wire BPSCCO using Ag sheath after sintering at 860 °C for 24 h in (a) First step process, (b) Second step process, (C) Third step process, and (d) Fourth step process

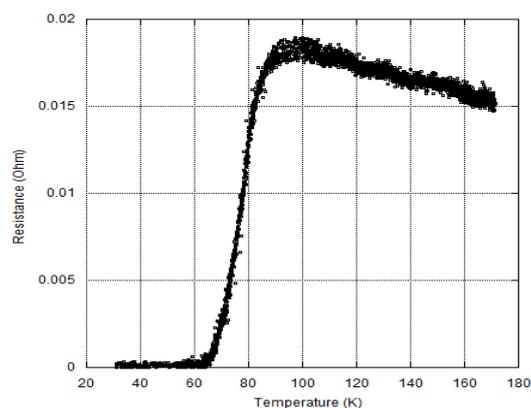


Figure 3. Resistance dependence on temperature for sample with third step

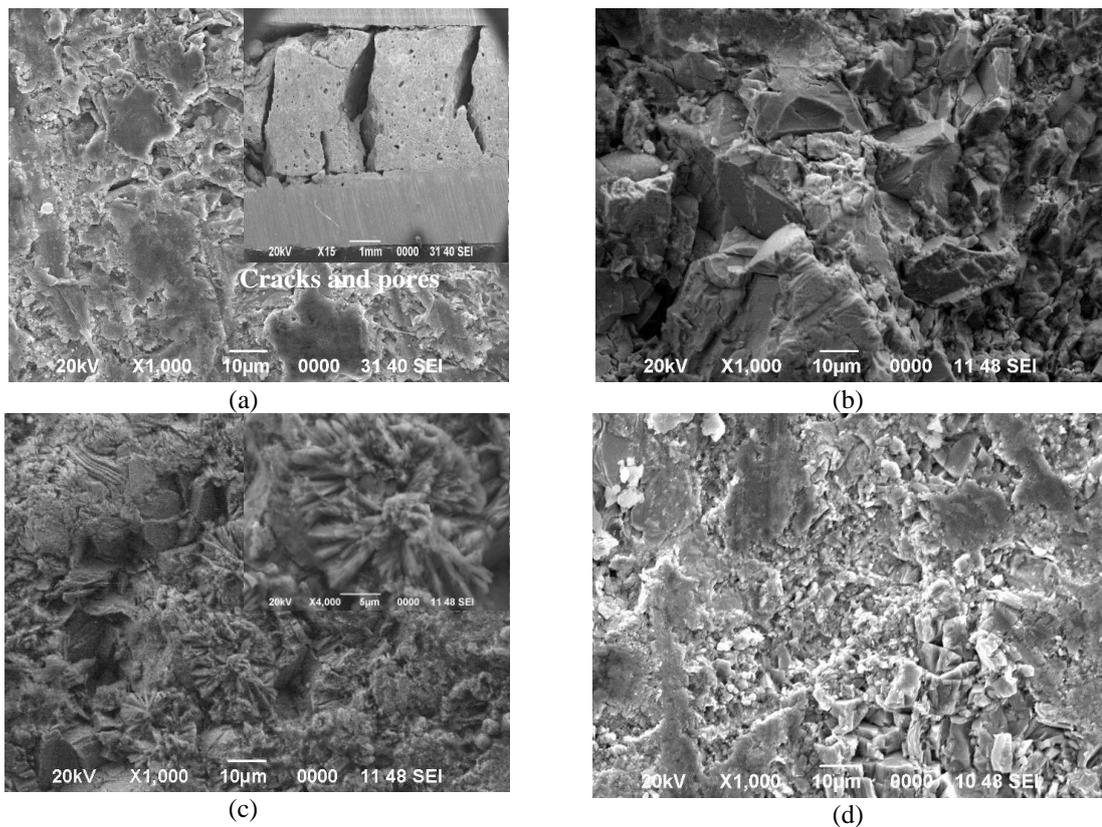


Figure 4. (a) The surface morphology of sample after first step, (b) The surface morphology of sample after second step, (c) The surface morphology of sample after third step, (d) The surface morphology of sample after fourth step

In Figure 3, the different microstructure and phase content observed in the scanning electron microscopy morphology of samples sintered in air atmosphere at 860 °C for 24 h are reflected in the $R-T$ curves. The four microstructures were taken from the sintered samples prepared under different step-rolling process. The sintered contained fine microstructure of deformed-shaped grains. The anisotropic grain shape is considered to originate from the deformed shape of the rolling process. In Figure 4a, the presence of crack and small voids appearing along the thickness of composite is noticed in the matrix. While, in Figure 4b, for sample after rolled at second pass, the small voids and cracks were decreased. In Figure 4c, the sample sintered in third pass rolling has changed the grain structure to BPSCCO 2212 flower-like grain with a length of about 10 μm. In Figure 4d, BPSCCO 2212 flower-like grains structure disappear with less pores both in size and population. The presence of BPSCCO 2212 flower-like grain formation in the sinter after rolled at third pass is quite interesting. This differs from the expectation that both the size and number of residual pores inside the monofilament tape decrease will enhance the textural growth of BPSCCO 2212-plate-like grains with traditional PIT process [10]. The population of the flower-like grain structure in the matrix is fairly uniform implies the larger effective current carrying areas (transport properties) of BPSCCO 2212, as is seen in Figure 2. Obviously, the decrease of both the size and population of residual pores played an important factor in highly textured BPSCCO 2212 and enhance the transport properties of BPSCCO 2212. This suggests that reaction of BPSCCO 2212 with Ag sheath play a crucial role in the suppression of BPSCCO 2212 formation during heat treatment. It is known that intimate contact of between BPSCCO 2212 and Ag sheath can be considered as instantaneous diffusion of which Cu dissolution in Ag at the BPSCCO/Ag interface to activate Ag-Cu-O solid solution is a thermodynamically favorable reaction [12]. Cu was considered as impurity because the purity of Ag used in this study was 92.5 %.

However, Cu in the Ag sheath in filament is considered beneficial to process kilometer length of BPSCCO 2212 superconducting wire. In order to check the reaction of BPSCCO 2212 into Ag sheath, more detailed study are required to provide clear understanding of this phenomenon.

4. Conclusions

Monofilament-wire silver sheathed BPSCCO superconductor has been successfully made. The XRD pattern of sample sintered in the first and second rolling process shows the traceable amounts of constituent oxide powders, while samples sintered in third rolling process shows superconducting phase BPSCCO 2212. The amount of superconducting phase in sample sintered in fourth rolling process is not traceable. Meanwhile, $T_{c,onset}$ and $T_{c,final}$ superconductivity (90 K and 70 K) is found after third pass rolling and heating process. As observed in SEM image, the superconducting BPSCCO 2212 flower-grain like shape is anisotropic due to rolling process. T_c was not found in first and second step due to small void and cleavage along the matrix.

Acknowledgments

This project has been supported by the fund of Indonesian Institute of Sciences (LIPI) in 2016

References

- [1] Sotelo A *et al* 2014 *Journal of the European Ceramic Society*
- [2] Awana V P S *et al* 2013 *Materials Chemistry and Physics* **139** pp 681–688
- [3] Lu T *et al* 2015 *Superconductivity and its applications* **519** pp 24–27
- [4] Cicek O, Yetis H and Gencer A 2014 *Cryogenics* **63** pp 143–147
- [5] Malozemoff A P 2013 *Physica C*
- [6] Darsono N *et al* 2015 *Supercond Nov Magn*
- [7] Patel R H, Nabialek A and Niewczas M 2005 *Supercond. Sci. Technol.* **18** pp 317–324
- [8] Pramono A W 2000 *Quantitative analysis of the thermomechanical properties of Cu-18wt%Nb in-situ metal matrix composite wire and the Cahn-Hilliard simulation of its thermal phase evolution* (Ph.D. thesis, RWTH Aachen)
- [9] Li Q *et al* 2001 *U.S. Patent No. 6,311,386 B1* (6 November 2001)
- [10] Zhang S *et al* 2015 *Physics Procedia* **65** pp 57–60
- [11] Kadar J *et al* 2016 *Supercond. Sci. Technol.* **29**
- [12] Weselowski D E *et al* 2005 *Supercond. Sci. Technol.* **18** pp 934–943