Bi2212 Superconducting Tubular Conductors Prepared by the Diffusion Process for Current Lead

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Abstract. Bi2212 HTS conical tubular conductors have been prepared by the diffusion process for current lead application. The Bi2212 HTS layers are synthesized through the diffusion reaction between a Sr-Ca-Cu oxide substrate and a Bi-Cu oxide coating with Ag addition. Highly oriented HTS diffusion layers about 150µm in thickness are formed around both outside and inside of the conical tube 34/29mm in outside/inside diameter at larger end, 24/19mm in outside/inside diameter at smaller end, and 100mm in length. The transport current has been measured using two cryocoolers between 12 K at cold end and 40-70 K at warm end under 0.5 T. The transport current of 1,000 A was stably passed for 10 minutes at 50 K. The joint voltage of cold end was as low as 30µV at 50 K during 1,000 A running. The low voltage results from the low contact resistance between Bi2212 grains and Ag precipitated on the surface. Heat leakage conducted through the specimen has been newly evaluated using heat flux meter by steady-state heat flow method.

1. Introduction

High temperature superconductors (HTS) can be synthesized through the diffusion process between two components in an appreciably shorter reaction time than in the conventional sintering process. A thick and homogeneous HTS layer of Bi2Sr2CaCu2O8 (Bi2212) is easily synthesized by the diffusion reaction between Sr-Ca-Cu oxide substrate and Bi-Cu oxide coating [1-2]. The Bi2212 HTS cylindrical tubes prepared by the diffusion process were found to be promising for current leads with large transport current and small heat leakage [3-4]. In the present article, the transport current performance and the structures of Bi2212 HTS conical tubular bulk conductor prepared by the diffusion process are reported. The conical shaped tube yielded larger transport current due to the larger cross-sectional area at warm end, and smaller heat leakage due to the smaller cross-sectional area at cold end in comparison with the cylindrical tubes previously reported [5-6]. The Bi2212 HTS conical tubes synthesized by the diffusion process are attractive for a current lead to be used in superconducting magnet system.

2. Experimental

The preparation procedure of Bi2212 conical tubular specimen through the diffusion process is schematically shown in Figure 1. The substrate is composed of Sr-Ca-Cu oxide with the composition
ratio of Sr:Ca:Cu=2:1:2 (referred to as “0212”). The calcinated 0212 oxide powder was formed into conical tube 34/29 mm in outside/inside diameter at the larger end, 24/19 mm in outside/inside diameter at the smaller end, and 100 mm in length by cold isostatic pressing (CIP). The tubular bulk was then sintered at 1000°C in open air. The coating is composed of Bi-Cu oxide with the composition ratio of Bi:Cu=2:1 (referred to as 2001). The calcinated 2001 oxide powder with 30 mass% Ag2O addition was mixed with wax to form slurry, and was coated around the conical tubular substrate. The diffusion reaction was performed at 850°C for 20 h to produce the Bi2212 HTS layer. Ag added to the coating precipitates on the surface of the specimen after the heat treatment. The Ag paste was coated around both ends of the conical specimen, and was sintered at 800°C to form the Ag contacts.

Figure 2 (a) shows the Bi2212 conical tubular specimens as finished in diffusion process for current lead. The Ag contacts of about 100 µm in thickness are formed around both ends of the specimen. Figure 2 (b) indicates the specimen soldered to both Cu end caps using commercial Sn-Pb solder to connect to Cu bus bar in measurement system for transport current. Four voltage taps were attached on the smaller and larger Cu caps (V1 and V4), and on the HTS (distance of 40 mm between V2 and V3). Five cernox resistance thermo sensors were attached on the smaller and larger Cu caps (T1 and T5), and on the HTS (distance of every 20 mm between T2, T3, and T4), respectively. A pair of SUS304 stainless steel boards serves as a shunt, and relieves thermal stress in the specimen.

Figure 3 shows a schematic illustration of measurement system [7] for transport current using two cryocoolers and a set-up of the specimen. Cu leads and the larger end (warm end) of the conical specimen are cooled through a 1-stage Gifford-McMahon (GM) cryocooler, and the smaller end (cold end) of the specimen is cooled through a 2-stage GM cryocooler in a vacuum cryostat. Resistive heaters are installed on the Cu leads and stages of cryocoolers to adjust the temperature of both end of the specimen. Magnetic field of 0.5 T is applied perpendicular to the specimen using a permanent magnet. The transport currents of the specimens are measured resistively by a dc four-probe method. The transport current density is obtained by dividing transport current by the cross-sectional area of Bi2212 HTS layers. The transport currents were measured in the facilities of Railway Technical Research Institute. The critical temperature Tc was measured resistively by a four-probe method. The structure of the prepared specimens was studied by an optical microscope (OM), scanning electron microscope (SEM), and X-ray diffractometry (XRD).

Figure 1. Preparation procedure of Bi2212 HTS conical tubes by the diffusion process.

Figure 2. Bi2212 conical tubular specimen. (a) as reacted, (b) connected to Cu end caps. Totally 4 voltage taps (V1-V4) and 5 cernox thermo sensors (T1-T5) were attached on the surface of HTS and Cu caps.
3. Results and discussions

3.1. Structural Aspects of Bi$_{2}$212 HTS Conical Tube

An optical micrograph taken on the outside and inside cross-section of the Bi$_{2}$212 conical tube specimen is shown in figure 4. The Bi$_{2}$212 diffusion layer of about 150-200 µm is synthesized around both sides of the substrate. Most of the Ag added to the coating precipitates on the surface of the Bi$_{2}$212 HTS layer after the diffusion reaction. The precipitated Ag of several 10 µm in thickness results in the low contact resistance between Bi$_{2}$212 HTS layer and Ag contact formed at both ends.

SEM micrograph taken on the fractured outside cross-section of the specimen is shown in figure 5. The diffusion layer is composed of thin plate-like grains grown along the diffusion direction, the radial direction of the conical tube. The characteristic structure of the diffusion layer results from the preferred grain growth along $a$-axis and $b$-axis direction in Bi$_{2}$212 HTS crystallization process.

Figure 6 shows the XRD patterns taken on the outside and inside surfaces of the conical tube specimen. A peak of the Ag is seen on the outside and inside surface after diffusion reaction. The XRD pattern indicates strong (200) peak in comparison with that of conventionally sintered Bi$_{2}$212 bulk surface after removing the Ag precipitation by NH$_{4}$OH/H$_{2}$O$_{2}$ etching solution. Some CuO

Figure 4. Optical micrographs taken on the outside and inside cross-section of the Bi$_{2}$212 specimen.
particles unsolved the etching solution remain on the surface. Therefore, the diffusion layer is found to
be composed of $a$-axis textured grains. The transport current longitudinally passes through the conical
specimen along the $a$-$b$ planes of the grains grown in the radial direction.

Figure 5. SEM micrograph taken on the fractured outside cross-section of the specimen.

Figure 6. X-ray diffraction patterns taken on the outside and inside surfaces of the specimen.

3.2. Transport Current Performance of Bi2212 HTS Conical Tube

Temperature dependence of transport current for the conical specimen at 0.5 T is shown in figure 7. The transport current was supplied at a ramp rate of 20 A/s. Arrows indicates that the transport current exceeds the capacity limit of 1,000 A for the power source. The critical temperature of transport current of 1000 A at 0.5 T is 57.4 K, which corresponds to the current density of 33 A/mm² for the Bi2212 HTS layers at the larger (warm) end. The transport current decreases with increasing temperature of the conical specimen, and is about 800 A at 60.2 K, 600 A at 62.5 K and 200 A at 70 K, respectively. Since the offset $T_c$ of the specimen is 78 K, almost no transport current passes at 77 K.

Figure 8 shows the transport current performance of 1,000 A for the Bi2212 specimen at 50 K and 0.5 T. The transport current of 1000 A at 50 K was stably run for 600 s with almost no voltage on the HTS part (between V2 and V3). The voltages of both joints increased with increasing transport current, and were 280 µV at the warm joint and 21 µV at the cold joint after reaching 1,000 A. Then, the voltages of warm and cold joints increased to 380 µV and 34 µV after holding the current for 600 s. Therefore, the heat load at cold joint is as small as about 30 mW due to the small Joule heat. The small heat load, that is, low voltage at cold joint results from low contact resistance between Bi2212 grains and Ag precipitated through the diffusion reaction.

Figure 7. Temperature dependence of transport current at 0.5T for the Bi2212 HTS specimen.

Figure 8. Transport current performance of 1000A for the Bi2212 HTS specimen at 50K and 0.5T.
Figures 9(a) and (b) show voltages of warm end (V3-V4) and cold end (V1-V2) versus transport current at warm end temperatures between 40K and 60K. The voltages at warm end increased with increasing transport current and temperature in figure 9(a). Above 50 K, the voltages increased sharply and non-linearly with transport current since resistivity of Cu increased by a factor of 4 from 40 K to 60 K [8] and joule heating was generated. On the other hand, the temperatures at cold end increased by 2 K, although the temperature at warm end increased from 40 K to 60 K. The voltages at cold end increased almost linearly with transport current to 20 µV at transport current of 1,000 A.

Figure 9. Voltages of warm end (a) and cold end (b) versus transport current at warm end temperatures between 40K and 60K.

3.3. Estimation of Heat leakage for Bi2212 HTS Conical Tube

Figure 10 shows a schematic illustration of measurement system for heat leakage conducted through the specimen using heat flux meter and a set-up of the specimen. The measurement system for transport current shown in figure 3 was modified to the system for measuring heat flow. Cu leads and the larger end of the conical specimen are similarly cooled through a 1-stage GM cryocooler, and the smaller end is cooled via the heat flux meter through a 2-stage GM cryocooler. The heat flux meter

Figure 10. Schematic illustration of measurement system for heat leakage using heat flux meter.

Figure 11. Heat leakage conducted through the specimen versus temperature at warm end. The temperature at cold end is held constant at 20 K.
made by Cu metal 8 x 2 mm in cross-section and 100 mm in length is installed between the specimen and 2-stage GM cryocooler. The heat flux is evaluated from cross-sectional area, length, temperature difference between higher end \((T_H)\) and lower end \((T_L)\) and thermal conductivity in the Cu-made heat flux meter by steady-state heat flow method with no transport current.

Heat leakage \(Q_c\) conducted through the specimen versus temperature at warm end is shown in Figure 11. The temperature at cold end is held constant at 20 K. The heat leakage \(Q_{mea}\) measured by heat flux meter increases almost linearly with increasing temperature at warm end, that is, temperature difference between warm end and cold end in the specimen. \(Q_{mea}\) at warm end of 60 K doubles in comparison with \(Q_{mea}=0.85\) W at 40 K. The modified measurement system using heat flux meter shown in figure 10 is useful for evaluating the heat flow conducted through the specimen with no cryogen such as helium and nitrogen.

The \(Q_c\) mainly consists of the heat leakage of the SrCaCu oxide substrate and precipitated Ag on the specimen. The sum of the heat leakage calculated from the substrate: \(Q_{cal(subs)}\) and the Ag: \(Q_{cal(Ag)}\) almost agreed with the measured one by heat flux meter. The heat leakage of \(Q_{cal(Ag)}\) is about 60% of the total heat leakage since the thermal conductivity integral of the Ag is a factor of 50-60 compared to that of the substrate. In the previous study [6], the conical tube 200 mm in length was successfully fabricated and high performances were evaluated. The longer tubes are effective at decreasing the heat leakage of the specimen. The twice-long-sized tube specimen reduces the heat leakage by one-half. Furthermore, removal of the precipitated Ag except both ends for current contacts using etching solution depresses extremely the heat leakage conducted through the specimen. The transport current of the specimen hardly falls by dissolving the precipitated Ag since the Bi2212 diffusion layer is not subject to the corrosion [2].

4. Conclusions
Bi2212 HTS conical tubular conductors have been prepared by the diffusion process for current lead. The highly oriented Bi2212 layers with 150 µm in thickness are synthesized around both outside and inside of the conical tube. The transport current of 1,000 A was stably passed for 10 minutes at 50 K, which corresponds to the current density of 33 A/mm². The transport current decreases with increasing temperature at the warm end, and is about 800 A at 60 K and 600 A at 62.5 K. The joint voltage of cold end was as low as 30 µV at 50 K during 1,000 A running. The low voltage results from the low contact resistance between Bi2212 HTS grains and Ag on the surface after diffusion reaction. Heat leakage conducted through the specimen was 0.85 W between 40 K at warm end and 20 K at cold end.

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