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CONTENTS

Bi-BASED MONO-CORE SUPERCONDUCTOR TAPES: FABRICATION PROCESS AND THE EFFECT OF THE THERMOMECHANICAL TREATMENT ON THE TRANSPORT PROPERTIES

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ABSTRACT

Fabrication of the Bi-based tapes started with the preparation of its precursor powder with composition of $Bi_{1.8}Pb_{0.4}Sr_2Ca_{2.2}Cu_3O_{10+y}$. The post-annealed powder with a majority of 2212 phase was then ground before it was packed into a silver tube. Then the composite was sway-rolled and drawn into wire with diameter of 1.08 mm. To make it into tape form, the wire was cut into shorter length before went through rolling process using a roller machine. The tapes were divided into sample groups and underwent different thermo-mechanical treatments. All the final processed Bi-based tapes had mixed superconductor phases of 2223 and 2212. Most part of HTSC grains in the tapes melted during the sintering. Transport voltage-current characteristics of the tapes was found to obey the power law relationship. The thermo-mechanical treatment was found necessary to improve the structural quality of the tapes.

Keywords: Bi-based tapes, powder-in-tube, thermo-mechanical treatment, superconductor

1. INTRODUCTION

Research activities and technology development of superconductor materials have shown a great advancement since the discovery of high temperature superconductor (HTSC) eighteen years ago. The direction of its application has become much clearer and wider especially in the powder application industry. Superconductors in the form of wires and tapes are the most desired as they can be used in transmission cables, transformers, utility generators and magnet[1, 2]. Due to the fact that HTSC is mechanically

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brittle, ductile medium is used to support HTSC before undergoing the fabrication process of the HTSC[3,4] wire.

Several methods have been introduced to produce HTSC wire but powder-in-tube (PIT) method is the most popular[3]. In general, PIT method consists of a series of processes, which starts with the calcinations of precursor powder, tubing, cold working (swag-rolling or drawing) and followed by repeated heat treatment and mechanical deformation (either rolling or pressing). The repeated heat treatment and mechanical deformation is also known as thermo-mechanical treatment.

In this paper, the fabrication process of mono-core Bi-based tapes using PIT method and the effect of the thermo-mechanical treatment on the transport properties of the tapes will be briefly discussed.

2. EXPERIMENTAL METHODS

2.1 Preparation of the Precursor Powder

The fabrication of the tapes can be divided into two parts. First is the preparation of the precursor powder for the tapes, and this was followed by the preparation of tapes itself. The precursor powder was prepared by solid-state reaction using high purity Bi_2O_3, PbO, SrO, CaO and CuO powders as raw materials. Initially, each powder was added separately according to the required amount in the following equation:

$$0.9Bi_2O_3 + 0.4PbO + 2SrO + 2.2CaO + 3CuO \longrightarrow Bi_{1.8}Pb_{0.4}Sr_2Ca_{2.2}Cu_3O_{10+y}$$

The powders were then mixed and ground in the agate mould for two hours. The fine mixed powder was calcined in air (~21% O_2) at a temperature of 810 °C for 24 hours. The heating temperature and period were chosen to produce precursor powder with 2212 phase as a majority phase[5]. The process was repeated twice in order to increase the powder homogeneity. The XRD pattern of the final form of the precursor powder was examined. Figure 1 shows the XRD patterns of the final form precursor powder. 2212 phase and 2201 phase are the major phases in the sample.

2.2 Preparation of the HTSC Tapes

The precursor was ground for two hours to produce a fine powder before it was put into a pure Ag tube with one end closed. The tube had an outer diameter of 6.03 mm and an inner diameter of 4.43 mm. The powder was packed into the tube until the packing density of the powder reached 3.14 g/cm^3. The opening end of the tube was then tightly closed. The Ag tube helps to shape HTSC and gives strength to withstand force during the elongation. Ag is a preferable element for the tube because it is inert toward HTSC material and it allows oxygen gas to diffuse through to reach HTSC during the heat treatment[3]. It is also found that it can enhance the growth of the 2223 phase of bismuth based HTSC (or BSCCO) [3].

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The composites were then swagged and drawn to a final diameter of 1.08 mm using motor-powered rolling and drawing machine. Both swag-rolling and drawing processes were set at a low rate to minimize the occurrence of defects in the wires. It was then rolled into tape form using a roller rotated at a rate of 0.6 m/min. Tape's thickness was reduced by 25% or less from the initial thickness for every deformation process to reduce the sausaging (wavier core/silver interface) on the tapes[6]. The deformation process continues until the thickness of the tape reaches 0.32 mm. The long tape was cut into 3 cm pieces. These pieces were divided into groups of sample #1, #2, #3 and #4 subjected to different thermo-mechanical treatments.

2.3 Measurements

The phase of the samples was identified by powder X-ray diffraction (XRD) method using a Siemens D 5000 diffractometer with Cu-Kα source. A Philips XL-30 scanning electron microscope (SEM) was used to observe the microstructures of the samples. Transport critical current density (Jc) was measured using the 1.0 μV/cm criterion and four probe method at 77 K in magnetic fields ranging from 0 to 0.5 T. The magnetic fields were applied perpendicular (**B⊥**) to the plane of the tapes. J_c was calculated by dividing the tape's critical current (I_c) with their corresponding superconductor core cross-sectional area. The correction for current (I) through superconductor core was made by substracting the current through Ag sheath from the total current obtained at higher applied field and higher current[7].

3. RESULTS AND DISCUSSION

Figure 1 shows the XRD pattern of the precursor powder used for HSTC fabrication. It is clear that 2212 phase and 2201 were the major phases in the precursor powder.

XRD patterns for all processed tapes are shown in Figure 2. All tapes showed the existence of mixed superconductor phases of 2223 and 2212. From the volume fraction of the 2223 phase (X_{2223}) (Table 1), it was observed that the amount of 2223 phase increased relatively with the increase of sintering period which is consistent with previous studies[3]. The molar fraction of the 2223 phase (X_{2223}) and 2212 phase (X_{2212}) in the examined tapes was determined by $I_{H(0010)}/[I_{H(0010)} + I_{L(008)}]$ and $0.5I_{L(008)}/[I_{H(0010)} + I_{L(008)}]$ where the $I_{H(0010)}$ and $I_{L(008)}$ were the intensity of (0010) peak at B⊥ =24.0 °C and (008) peak at B⊥ = 23.2[8]. SEM photo for the sample #3 (Figure 3) showed clearly that most part of HSTC grains in the tapes was melted during sintering as compared to the SEM photo of the precursor powder grains used in tape fabrication as shown in Figure 4.

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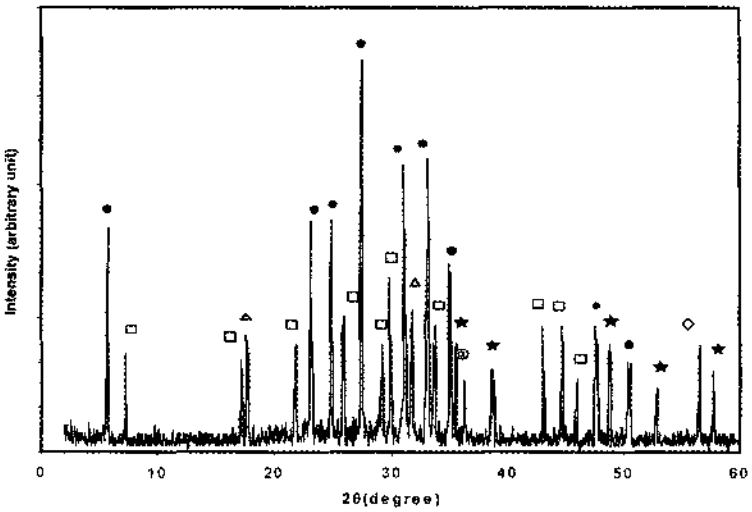

Figure 1: XRD pattern for the precursor powder used for HTSC fabrication.

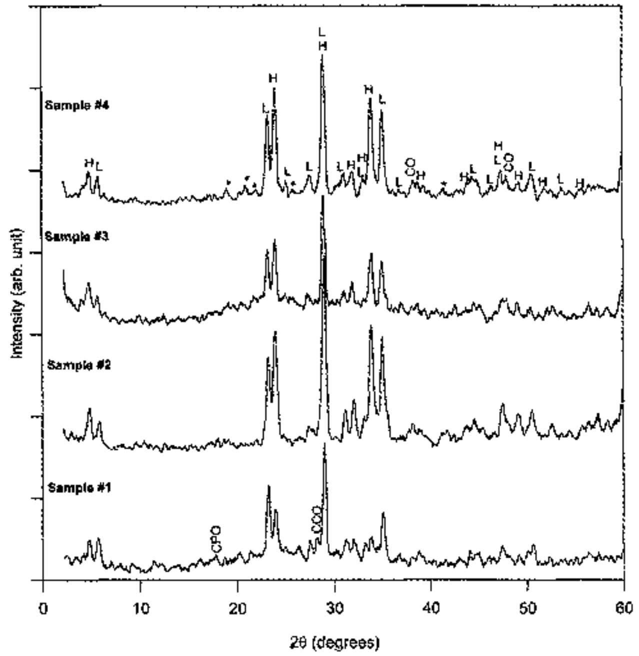

Figure 2: XRD patterns for sample #1, #2, #3 and #4. H:2223 phase, L:2212 phase, CCO:Ca_2CuO_3, CPO:Ca_2PbO_4, CO:CuO, *: unknown phases.

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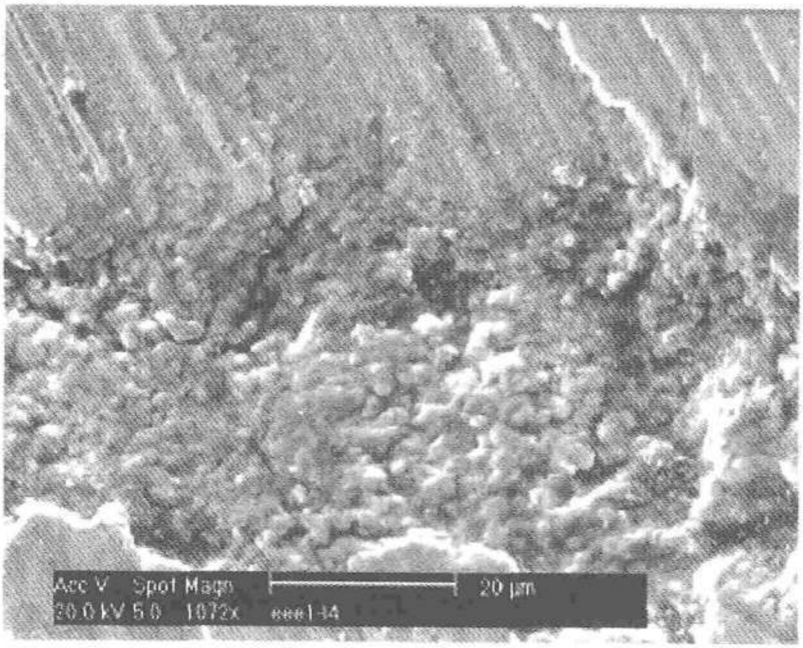

Figure 3: SEM photo of sample #3 in longitudinal sectional view after thermo-mechanical treatment.

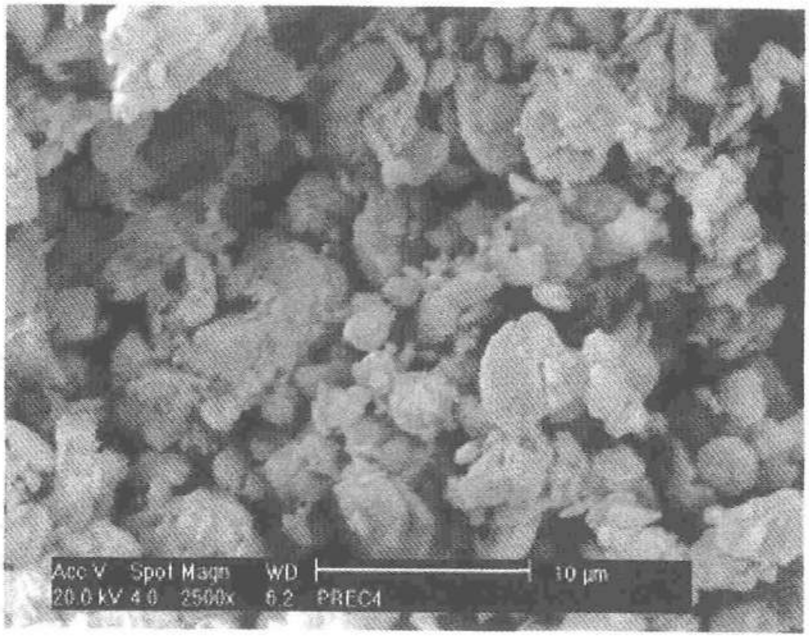

Figure 4: SEM photo of the precursor powder grains used in tape fabrication.

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Transport electric field-current (E-I) characteristics of our tapes in various magnetic fields B are well described by powder law relationship, $E\text{-}I^n$. The exponent n can be derived from curve fitting on the E-I characteristics of each samples in applied field from 0 T to 0.10 T. Figure 5 shows the E-I characteristics of sample #1 in log-log scale. The ratio of J_c/n reflects the structural quality of tapes from the aspect of effective number of intergrain connections (supercurrent links) and pinning centres. Higher values of Jc/n indicate better structural quality[9]. Figure 6 shows that J_c/n for sample #2 is the highest and therefore has the best structural quality followed by #3, #4 and #1.

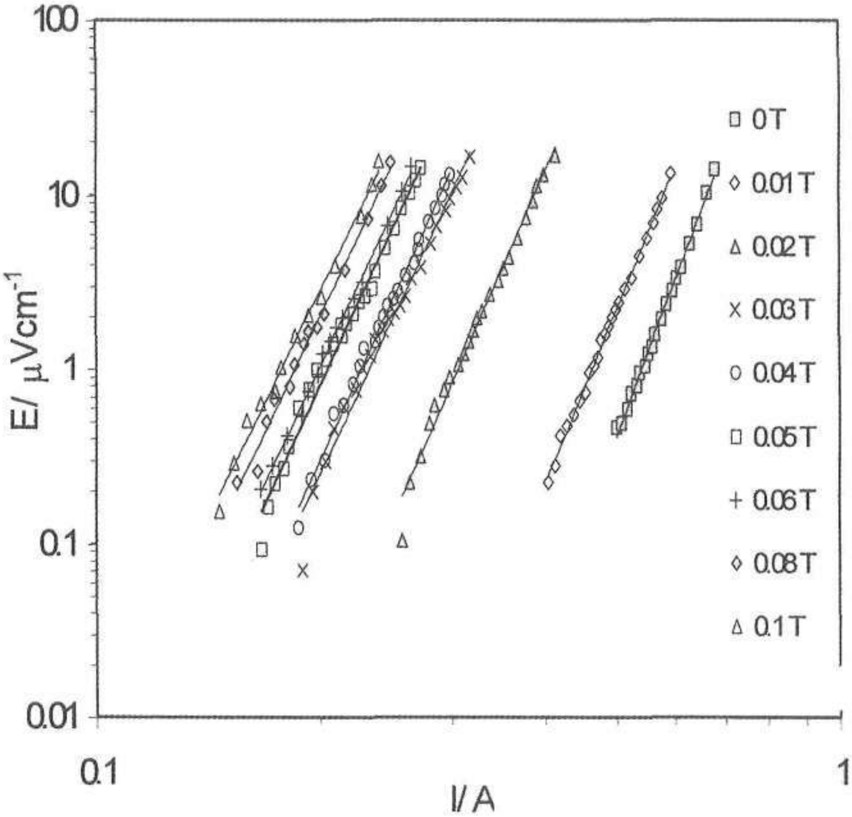

Figure 5: *E-I* characteristics of sample #1 at 77 K in applied field from 0 – 0.10 T.

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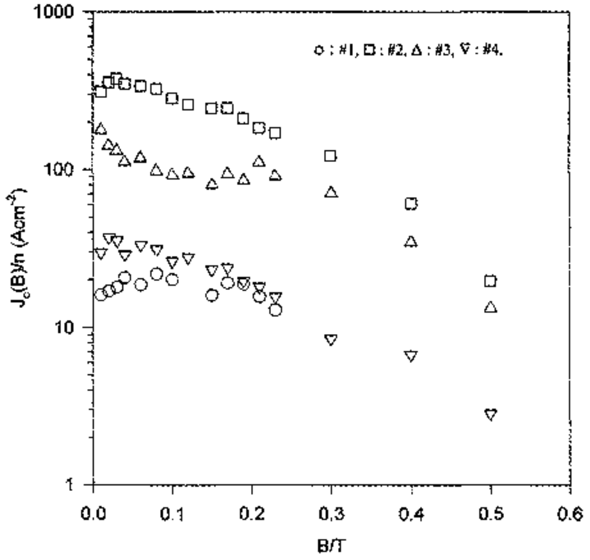

Figure 6: Ratio of J_c/n versus applied B ⊥ to the tape plane of the samples at 77 K.

It clearly shows that the thermo-mechanical treatment with one intermediate rolling improved the structural quality of tapes. It is found that the existence of unhealed micro-cracks produced during the second intermediate rolling caused the quality of tapes to deteriorate. This may due to an insufficient amount of liquid phase which consists of Bi, Pb, Ca, Sr, and Cu elements that existed between the superconding grains to heal the cracks[10]. However, structural qualities of tapes prepared without intermediate rolling (sample #4) were found to be lower than the tapes prepared with intermediate rolling (sample #2 and #3). This shows that thermo-mechanical treatment generally improves the structural quality of the tapes.

In order to improve the structural quality of the tapes, it is suggested that the composition of tape's precursor powder should have a majority of 2212 phase[11] which is more reactive, compared with a mixture of 2212 and 2201 phase. Reactivity of the precursor powder is crucial to produce liquid phase, an important agent to heal mechanical deformation induced cracks and formation of 2223 phase supercurrent links. It is also necessary to improve the rolling process of the tapes in order to reduce the formation of microcrack. Factors such as diameter and rotating speed of the roller, percentage of thickness reduction etc., need to be considered in order to decrease the stress applied on the tapes during rolling.

4. CONCLUSION

Bi-based mono-core tapes were fabricated using powder-in-tube (PIT) method. The final form of Bi-based tapes

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had mixed superconductor phases of 2223 and 2212. Most parts of HSTC grains in the tapes melted during the sintering process. Transport voltage-current characteristics of the tapes obeyed the power law relationship. In general, the thermo-mechanical treatment was found necessary to improve the structural quality of the tapes.

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