Solid State Science and Technology, Vol. 15, No 1 (2007) 43-48
ISSN 0128-7389

FABRICATION AND TRANSPORT PROPERTIES OF In-DOPED Tl1212/Ag SINGLE-CORE DIP-COATED TAPES

M. I. Yusof[a], R. Omar[a] A. K. Yahya[a] and Z. Awang[b]

[a]*Faculty of Applied Sciences, Universiti Teknologi MARA,40450 Shah Alam, Selangor*
[b]*Microwave Center, Faculty of Electrical Eng,*
Universiti Teknologi MARA, 40450 Shah Alam, Selangor.

ABSTRACT

Superconducting powder from high purity chemicals with nominal composition of $Tl_{0.9}Bi_{0.1}Sr_{1.9}In_{0.1}Ca_{0.9}Y_{0.1}Cu_2O_7$ were used to fabricate Tl-1212/Ag superconducting tapes using the dip-coating (DC) method. The tapes were subjected to reannealing under different heating conditions where some of the tapes were subjected to intermediate mechanical rolling. Results showed that annealing temperature and annealing duration together with intermediate mechanical rolling (IR) could be optimized to increase T_c and 1212 phase formation and J_c of In-substituted Tl-1212/Ag tapes. The highest J_c was observed for the tape, which was reannealed at 910 °C for a total duration of 60 minutes and subjected to IR. The increased J_c could be due to the densification of superconducting core and increase in 1212 phase after thermomechanical treatment.

INTRODUCTION

The phenomenon of superconductivity has always been very exciting and has been explored both for fundamental and scientific interest and for possible technical applications. The most common method used for fabricating long length superconducting wire is the oxide powder-in-tube (PIT) method where the oxide powder is placed inside the silver tube and then rolled and swaged into wire [1,2]. Unlike the PIT process, the dip-coating (DC) process is a simpler method and does not require any mechanical deformation steps for fabricating tapes. In the DC process, a tape sample is fabricated by simply dipping Ag strip continuously through slurry made of oxide powder and organic solution. While the PIT process has become a main method for Bi-2223, dip-coating (DC) process has become a main method for fabricating long length Bi-2212 tape conductors. So far, J_c up to ~5 x 10^5 Acm^{-2} at 4.2 K, 10 T has been obtained from short Bi-2212 tape samples made by the DC process [3,4]. Dip-coating (DC) process was reported as effective for obtaining highly oriented superconducting grains with high J_c value at 4.2 K in high magnetic fields above 20 T [3,4]. One of the advantages in processing the Tl-based tapes is the relatively short annealing time compared with Bi-based tapes [5]. However, fabrication of Tl-based tapes using the dip-coating (DC) technique is not as extensively reported for compared to Bi-based tape.

In this paper, we report synthesis of Tl-1212 dip-coated tapes with nominal composition of $Tl_{0.9}Bi_{0.1}Sr_{1.9}In_{0.1}Ca_{0.9}Y_{0.1}Cu_2O_7$ prepared from solid state reaction method as a starting material before dip coating process. The effects on superconducting properties

Corresponding Author: mohdi113@salam.uitm.edu.my

of dip-coated tapes when subjected to different annealing temperature were investigated. The results from dc electrical resistance measurements, X-ray powder diffraction spectra (XRD) and the scanning electron micrographs (SEM) are presented. The effect of thermo-mechanical treatments on T_c of the tapes in zero field and low magnetic fields up to 0.8 T are presented and discussed.

EXPERIMENTAL DETAILS

Sample with nominal composition of $Tl_{0.9}Bi_{0.1}Sr_{1.9}In_{0.1}Ca_{0.9}Y_{0.1}Cu_2O_7$ were prepared by conventional solid-state reaction method from high purity (≥99.99%) oxides of $SrCO_3$, In_2O_3, CaO, Y_2O_3 and CuO. The preparation method for the superconductor material is described in ref [6]. In order to make the slurry, the superconducting powder was then mixed with an organic solution made of solvent (trichloroethylene), organic binder (polyvinyl butyral) and dispersant (sorbitan trioleate) with appropriate ratios [7] to have proper viscosity. Then the slurry was stirred for 3 - 4 hr to obtain a homogeneous suspension before DC. Ag strips of size 4 mm x 20 mm with thickness of 0.03 mm were attached to the dipping sticks and then repeatedly dipped into the slurry to prepare single sided dip-coated Ag strips. After drying at room temperature, the coated strips were left dried at 80^{o}C for 48 hours and then the tape was wrapped with 0.03 mm thick pure Ag foil and heated at 450 °C in air for 3 hours to burn and remove organic materials. After heating, the tape samples were annealed between 800 ^{o}C - 910 ^{o}C in flowing oxygen and furnace cooled to room temperature. Some of the samples were subjected to intermediate rolling between two annealing segments in order to study the effect of the mechanical treatment of the tape.

Powder X-ray diffraction analysis was carried out using a Bruker D8 Advance diffractometer with Cu-K radiation. For Tl1212 tape, the top layer of the tape was peeled off and the tapes were sent for XRD. Volume fraction of all samples for any particular phases was determine from the ratio of the highest XRD intensity peak for the phases to the total intensity peak (highest peak only) of all phases under consideration. The electrical resistance measurements between 16 K and 300 K were carried out using the four-point-probe method with silver paint contacts in a Janis model CCS 350ST cryostat in conjunction with a closed cycle refrigerator from CTI Cryogenics model 22. A reflected optical micrograph model Citovel 2 was used to determine the cross sectional area of the tapes while scanning electron micrographs to study the microstructure of the tapes were recorded using JEOL JSM Model 6360LA scanning electron microscope. J_c measurement was performed on all tapes using electrical field's criterion of 1μV cm^{-1} to define J_c. The transport current density measurements were made in zero and applied fields up to 0.80 T in a direction perpendicular and parallel to the tape plane.

RESULTS AND DISCUSSION

Powder X-ray diffraction patterns for all bulk and tape samples showed major 1212 phase with tetragonal unit cell (space group P4/*mmm*) and minor 1201phase. Table 1 shows thermomechanical history with $T_{c\ zero}$, $T_{c\ onset}$, thickness and critical current

Corresponding Author: mohdi113@salam.uitm.edu.my

density and 1212 phase formation for In-substituted tapes I1 to I8.

Table 1: Thermomechanical history with $T_{c\ zero}$, $T_{c\ onset}$, thickness, current density J_c and 1212:1201 volume ratios for In-substituted tapes. (IR= Intermediate Rolling)

Sample	Tape processing	T_c zero (K)	T_c onset (K)	Thickness (mm)	J_c at 20 K (A/cm^2)	1212:1201 (volume %)
I1	910 ^oC / 20 min + 910 ^oC /40 min	74	93	0.11	520	68:32
I2	870 ^oC / 20 min + 870 ^oC /40 min	77	96	0.14	330	66:34
I3	820 ^oC / 20 min + 820 ^oC /40 min	22	38	0.17	450	63:37
I4	800 ^oC / 20 min + 800 ^oC/40 min	43	51	0.12	210	61:39
I5	910 ^oC/20 min + IR + 910 ^oC/40 min	75	85	0.09	880	81:19
I6	870 ^oC/20 min + IR + 870 ^oC/40 min	51	72	0.08	410	62:38
I7	820 ^oC/ 20min + IR + 820 ^oC/40 min	69	88	0.08	410	63:37
I8	800 ^oC/20min + IR + 800 ^oC/40 min	53	75	0.08	340	67:33

To study the effect of re-annealing on T_c and J_c, tapes I1, I2, I3 and I4 were re-annealed at 800^{o}C, 820^{o}C, 870^{o}C and 910^{o}C respectively for 40 minutes after the first annealing segment. Our results generally showed that re-annealing tapes I1, I2, I3 and I4 causes $T_{c\ zero}$ of the tapes to decrease with decreasing re-annealing temperature. High T_c above 70 K was recorded from samples re-annealed above 870^{o}C. Results on XRD diffraction (Tab.1) shows that 1212 phase is the major phase for all the tapes. Our results show that generally J_c value and 1212 volume percent increase with increasing annealing temperature. The highest J_c value of 520 Acm^{-2} and $T_{c\ zero}$ of 74 K was observed for tape I1 annealed at 910^{o}C. From these results, we suggested that annealing at high temperature improves 1212 phase formation and transport J_c.

To study the effect of intermediate rolling on T_c and J_c , tapes I5, I6, I7 and I8 were rolled after annealing at 910^{o}C, 870^{o}C, 820^{o}C and 800^{o}C respectively. The rolled tapes were then re-annealed for 40 minutes. Our result showed that the $T_{c\ zero}$ for all rolled tapes generally increased compared to non-rolled tapes except for I6 (Tab.1). J_c for rolled tapes was found to increase with increasing annealing temperature above 870^{o}C annealing temperature. Our results show that J_c for I5 which was rolled and re-annealed at 910^{o}C showed large a increase in J_c (880 Acm^{-2}) compared to unrolled tape I1 (520 Acm^{-2}) re-annealed at the same temperature. In addition, the 1212 phase formation in the tape I5 (81 volume %) is also higher that of tape I1 (68 volume %). Application of

Corresponding Author: mohdi113@salam.uitm.edu.my

intermediate rolling and re-annealing caused $T_{c\ zero}$ to increase for I5, I6, I7 and I8 compared to non-rolled samples. Rolling is expected to cause densification of the superconducting core and formation of microcrack inside the core. Re-annealing at 910°C immediately after rolling reconnects the grains and restores superconducting properties of the tapes. Fig.1 shows the J_c versus temperature curve for rolled and unrolled tapes. It clearly shows that J_c for tape I5 is much enhanced compared to the other tapes.

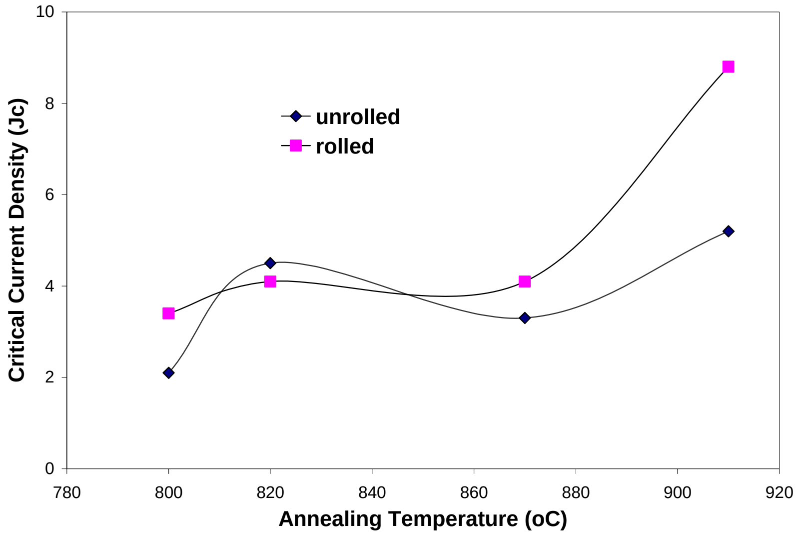

Figure 1: J_c (at 20 K) versus annealing temperature curve for all tapes (The solid lines are the guide to the eye only)

SEM micrographs for I1, I5 and I8 are shown in Fig. 2. Microstructure for tape I5 shows more compact and melted microstructure compared to tape I1. This is may be due to the application of intermediate rolling applied to tape I5. SEM micrograph for tape I8 revealed microcrack inside the core which may impede supercurrent flow in the oxide core [5,8]. However microstructure of tape I1 and I5 did not show any microcracks. This indicates that the microcracks are introduced in the tape as a result of mechanical rolling. Re-annealing at low temperature around 800°C is not effective to reconnect the cracks inside the core and results in lower J_c.

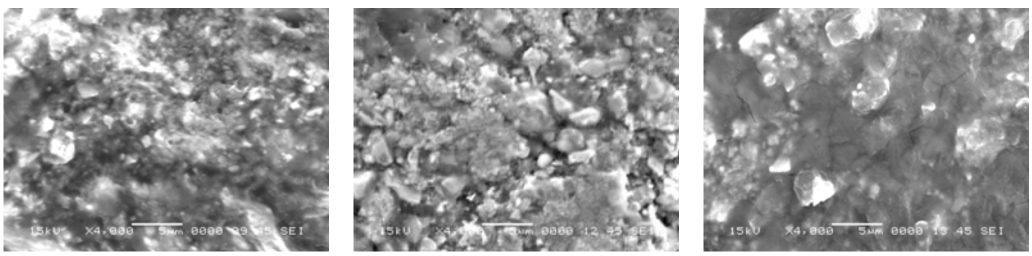

Figure 2: Microstructural view for tapes I1, I5 and I8

Corresponding Author: mohdi113@salam.uitm.edu.my

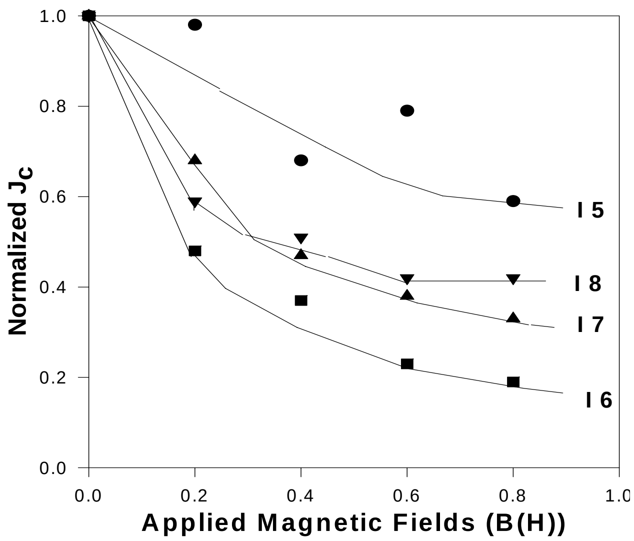

Figure 3: Normalized J_c (at 20 K) versus applied magnetic fields perpendicular to the plane for tapes I5, I6, I7 and I8 (The solid lines are the guide to the eye only)

Fig.3 shows the J_c versus applied magnetic fields for rolled tapes I5, I6, I7 and I8 oriented perpendicular to the tape's plane. An initial rapid suppression of J_c was observed for tape I6, I7 and I8 attributed to the weak-links phenomenon as observed in other polycrystalline high-T_c materials with random grain orientation. However, at higher fields (B>0.2 T) the J_c curve displays a plateau-like behavior which can attributed to the existence of strong-links network percolation path in the tapes [9]. Even though the J_c value found in this study is not encouraging, with the highest J_c value of only 880 Acm^{-2} , it is comparable to the J_c observed from J_c values reported by Chen et. al. [1] for rolled tapes $TlCr_{0.15}Sr_2Ca_{0.9}Pr_{0.1}Cu_2O_7$ rolled tapes using PIT method. The relatively low J_c of the dip-coated tapes can be attributed to the microstructure of the tapes where SEM micrographs of all tapes show no significant grains alignment with irregular shape grains. We believe that J_c can be further enhanced for Tl1212/Ag tape if a suitable chemical composition together with suitable thermomechanical treatment can produce long range directional grains alignment that will facilitate current flow in the superconducting core.

CONCLUSION

The effect of different thermo-mechanical treatments on Tl-1212/Ag tapes prepared from $Tl_{0.9}Bi_{0.1}Sr_{1.9}In_{0.1}Ca_{0.9}Y_{0.1}Cu_2O_7$ nominal composition was studied. We found that a proper combination of annealing temperature and intermediate rolling is necessary to increase J_c and phase formation in Tl1212 tapes. The highest J_c and 1212 phase formation was observed for tape I5 which was annealed at 910°C with intermediate rolling. Generally, the performance of J_c of the tapes in external magnetic fields (≤ 0.2 T) showed existence of weak links at low fields (≤ 0.2 T) followed by strong links which are dominant at higher fields.

Corresponding Author: mohdi113@salam.uitm.edu.my

Solid State Science and Technology, Vol. 15, No 1 (2007) 43-48
ISSN 0128-7389

REFERENCES

[1]. S.K.Chen, K.T.Lau, R.Shukor. (2002); *Material Science and Engineering* **B90**, 234
[2]. K.T. Lau, S.K Chen, R. Abd. Shukor. (2002); *PHY C* **377**, 547-552
[3]. H. Kumakura, K. Tagano, D.R Dietdrich, H. Maeda, J. Kase, T. Marimoto. (1991); *IEEE Trans. Magn* **27**, 1250
[4]. H. Kumakura, H. Kitaguchi, K. Togano, H. Maeda, J. Shimoyama, T. Marimoto, K. Namura, M. Seida. (1992); *Crogenics* **32**
[5]. D.E. Peterson, P.G. Wahlbeck, M.P. Maley, J.O. Willis, P.J. Kung, J.Y. Coulter, K.V. Salazar, D.S. Phillips, J.F. Bingert, E.J. Peterson and W.L. Hults. (1992); *PHY C* **199**, 161
[6]. A.K. Yahya, B.Musa and M.H.Jumali. (2005); *International Conference on Material for Advanced Technologies , Singapore*
[7]. Zhang, W., Pashitski, A., and Helltsrom. (1993); *E.E., Inst. Phys. Proceeding Superconductivity and Its Application* **273**, 599
[8]. V. Selvamanickam, T. Finkle, K. Pfaffenbach, P. Haldar, E. J. Peterson, K. V. Salazaar, E. P. Roth, J. E. Tkaczyk. (1996); *Physica C* **260**, 313
[9]. Kung P J, Wahlbeck P G, McHenry M E, Maley M P and Peterson D.E. (1994); *Phy C* **220**, 310

Corresponding Author: mohdi113@salam.uitm.edu.my