압연제조된 $Tl_{0.8}Pb_{0.2}Bi_{0.2}Sr_{1.8}Ba_{0.2}Ca_{2.2}Cu_3O_x$/Ag 선재에서의 높은 J_c

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High J_c's in just-rolled $Tl_{0.8}Pb_{0.2}Bi_{0.2}Sr_{1.8}Ba_{0.2}Ca_{2.2}Cu_3O_x$/Ag tapes

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Abstract – The grain morphology, the changes in morphology and Jc with the thermo-mechanical treatment (TMT) history, the field dependence of Jc and the nature of intergranular bonding were studied in $Tl_{0.8}Pb_{0.2}Bi_{0.2}Sr_{1.8}Ba_{0.2}Ca_{2.2}Cu_3O_z$/Ag tapes. As a result, incorporation of intermediate rolling during the final heat-treatment resulted in enhanced connectivity and directional alignment of plate-like Tl-1223 grains, and thus enhanced Jc. Jc's near 2.5×104 A/cm2 at 77 K and 0 T were obtained in just rolled tapes with an excellent reproducibility. The high Jc's seem to result from relatively easy recovery of excellent grain-connectivity during final heat-treatment after inter-mediate rolling, probably due to retarded Tl evaporation and excessive Ca content in the present composition. The strong field dependence of Jc even in low fields, however, indicated that there still existed significant weak-links and the degree of directional grain-alignment was far from the desired one. The intergranular binding in the tapes seemed to be mainly dominated by SIS junctions.

1. Introduction

It has been known that Tl-1223 super-conductor has an irreversibility line which is located at a relatively high position in H-T space, compared to those of other high T_c superconductors [1]. Therefore, it has been expected that Tl-1223 wires with high J_c at relatively high fields and tem-peratures will be able to developed and replace the present-aiming 20 K technolo-gy resulting from Bi-based superconduct-ing wires by 40 K technology.

Different from remarkable advances [2] in aspects of J_c and preparation cost in Tl-1223 film-type tapes prepared by so-called open methods, the progress in preparation of Tl-1223 tapes with high J_c's by the powder-in-tube (PIT) method has been slowed down. Tl-1223 tapes prepared by the PIT method have revealed relatively low J_c's due to poor grain-connectivity and little directional grain-alignment [1,3], although various chemi-cal compositions and preparation methods have been tried [3-9]. The J_c values have primarily depended on the nominal compo-sitions used for the tape cores. 2.1×10^4 A/cm^2 was obtained in $Tl_{0.93}Bi_{0.22}Sr_{1.6}Ba_{0.4}$-$Ca_2Cu_3O_z$ composition [7] as a highest reproducible J_c at 77 K and 0 T among pressed tapes, and 1.8×10^4 A/cm^2 in $Tl_{0.8}Pb_{0.2}Bi_{0.2}Sr_{1.6}Ba_{0.4}Ca_2Cu_3O_z$ [9] among just-rolled tapes, which are more import-ant in practical applications.

The present authors have tried several chemical compositions to overcome the poor connectivity and little directional grain-alignment in Tl-1223 tapes [9,10, 11]. As a result of such efforts, they could prepare just-rolled Tl-1223/Ag tapes with much enhanced grain-connectivity and directional grain-alignment, and thus high J_c's near 2.5×10^4 A/cm^2 at 77 K and 0 T by using a $Tl_{0.8}Pb_{0.2}Bi_{0.2}Sr_{1.8}Ba_{0.2}Ca_{2.2}Cu_3O_z$ nominal composition. In the present study, the grain morphology, the changes in mor-phology and J_c with the thermo-mechanical treatment (TMT) history, the field dependence of J_c and the nature of intergranular bonding in the tapes are presented.

2. Experimental

Tl-1223/Ag tapes of $Tl_{0.8}Pb_{0.2}Bi_{0.2}Sr_{1.8}$-$Ba_{0.2}Ca_{2.2}Cu_3O_z$ were prepared using the power-in-tube method incorporating an in-situ reaction method. The details on preparation of the tapes were shown in elsewhere [12].

J_c's of the tapes were evaluated using dc four-probe electrical measurements after immersing them into liquid nitrogen. The phases present in the tape cores were investigated using a Philips PW 1710 x-ray diffractometer (XRD) using a Cu K_α target. The XRD measurements were per-formed after pealing off the Ag-sheath on one side using a blade. The microstructure of the tapes was examined using a Hitachi S-2700 scanning electron microscope (SEM). The microstructures of the tape cores were examined after pealing off the Ag-sheath on one side using a blade. Magnetization was measured in fields up to 6 T parallel to the tape surface, by using a Quantum Design model MPMS superconducting quantum interference de-vice (SQUID) magnetometer. The field dependence of transport J_c was evaluated at 77 K in fields up to 1 T perpendicular to the tape surface and the current flow direction. The field was applied using a Lake Shore EM4-HV electromagnet. Ac susceptibilities were measured using Lake-shore 7000 ac susceptometer in ac fields of 1 kHz in a range of 0.01-19 Oe parallel to the tape surface.

3. Results and discussion

Table 1 shows variations of I_c and J_c of as-prepared tapes with the TMT history. The tapes which were totally heat-treated for about 7.5 h showed J_c's comparable to those of $Tl_{0.8}Pb_{0.2}Bi_{0.2}Sr_{1.6}Ba_{0.4}Ca_2Cu_3O_z$ tapes [9].

Table 1. Variations of I_c and J_c at 77 K and 0 T with the history of the thermo-mechanical treatment in as-prepared tapes.

Thermo-mechanical treatment history	Thick-ness (mm)	I_c (A)	J_c (A/cm^2)
820℃/20min+R+830℃/7h+R+830℃/7h	0.113	16.19	15,900
830℃/20min+R+820℃/7h	0.102	14.30	15,300
830℃/20min+R+830℃/7h	0.102	17.04	17,700
830℃/20min+R+830℃/7h+R+830℃/7h	0.108	11.12	11,300
830℃/20min+R+840℃/7h	0.125	14.28	12,800
830℃/20min+R+845℃/7h	0.115	14.60	14,200
830℃/20min+R+850℃/7h	0.119	12.65	12,000
840℃/20min+R+820℃/7h	0.108	15.52	15,600
840℃/20min+R+830℃/7h	0.112	16.67	16,600
840℃/20min+R+830℃/7h+R+830℃/7h	0.109	14.40	14,900
840℃/20min+R+830℃/7h+R+840℃/7h	0.098	18.79	20,900
840℃/20min+R+840℃/4h	0.125	12.35	11,800
840℃/20min+R+840℃/6h	0.114	16.45	15,900
840℃/20min+R+840℃/7h	0.112	16.92	16,700
840℃/20min+R+840℃/8h	0.119	17.19	15,900
840℃/20min+R+840℃/10h	0.139	13.74	11,900
840℃/20min+R+840℃/15h	0.151	11.61	9,300
840℃/20min+R+840℃/20h	0.141	13.08	11,100
840℃/20min+R+845℃/7h	0.114	16.72	16,300
840℃/20min+R+850℃/7h	0.131	14.87	12,500

It is worthwhile to note in Table 1 that upon incorporating an intermediate rolling during the final heat-treatment (HT) for 14 h, the J_c did not severely drop, but more or less increased. This behavior is quite different from that in the $Tl_{0.8}Pb_{0.2}$-$Bi_{0.2}Sr_{1.6}Ba_{0.4}Ca_2Cu_3O_z$ tapes [9]. So, the change in J_c with the TMT history, when intermediate rolling during the final HT was incorporated, was investigated. As a result, high J_c's near 2.5×10^4 A/cm^2 at 77 K and 0 T were obtained with excellent reproducibility in four tapes shortly heat-treated, rolled, heat-treated at 840℃ for 4 h, rolled and heat-treated again for 4 h, as shown in Table 2.

Table 2. Reproducibility of I_c, J_c and engineering critical current density (J_e) obtained in four tapes heat-treated at 840℃ for 20 min, rolled, heat-treated at 840℃ for 4 h, rolled and heat-treated again at 840℃ for 4 h. Each time, two tapes were prepared.

Trial no.	Thickness (mm)	I_c (A)	J_c (A/cm^2)	J_e (A/cm^2)
1	0.090	21.81	24,900	6,200
	0.095	21.70	23,500	5,900
2	0.090	21.23	25,200	6,300
	0.096	20.90	23,400	5,900

Fig. 1 shows XRD patterns taken in (a) a tape was heat-treated at 840℃ for 20 min, rolled and heat-treated at 830℃ for 7 h, (b) a tape prepared by rolling the tape (a) and heat-treating again at 840℃ for 7 h, and (c) a tape heat-treated at 840℃ for 20 min, rolled, heat-treated at 840℃ for 4 h, rolled and heat-treated again for 4 h. Most peaks in Fig. 1 corres-pond to Tl-1223 phase. The distinctions of Fig. 1 from XRD patterns in tapes of other compositions are as follows: First, incorporation of the intermediate rolling resulted in a significant increase in inten-sities of (00ℓ) peaks, as recognized by comparing Fig. 1(a) to Fig. 1(b) and 1(c). Such prominent (00ℓ) peaks shown in Fig. 1(b) and 1(c) indicates that Tl-1223 grains in the tapes are aligned in a pre-ferred orientation in a certain degree, and have not been reported in just-rolled Tl-1223 tapes of other compositions. They were only observed in a polycrystalline bulk of $Tl_{0.93}Bi_{0.22}Sr_{1.6}Ba_{0.4}Ca_2Cu_3O_z$ compo-sition pelletized using a very high pres-sure and then heat-treated [13]. It is estimated that a much lower pressure was applied during rolling in the present study. Therefore, Fig. 1 implies that the intermediate rolling is very effective in directional grain-alignment in the present composition. Second, $(Sr,Ca)_{14}Cu_{24}O_x$ peak intensity, which appeared to much increase after intermediate rolling and subsequent HT in $Tl_{0.8}Pb_{0.2}Bi_{0.2}Sr_{1.6}Ba_{0.4}$-$Ca_2Cu_3O_z$ tapes [9], did not noticeably increase in Fig. 1(b). As this peak appear-ed as an strong evidence of decomposition of Tl-1223 phase resulting from severe Tl evaporation during the subsequent HT [9, 14], non-prominence of this peak shown in Fig. 1(b) may indicate that the decompo-sition of Tl-1223 phase is less severe in the present composition than in $Tl_{0.8}Pb_{0.2}$-$Bi_{0.2}Sr_{1.6}Ba_{0.4}Ca_2Cu_3O_z$ tapes, and be a strong evidence for J_c increase in the tape heat-treated for 14 h with an intermediate rolling shown in Table 1, as will be discussed later. Third, $BaPbO_3$ peak, which appeared strongly in $Tl_{0.8}Pb_{0.2}Bi_{0.2}$-$Sr_{1.6}Ba_{0.4}Ca_2Cu_3O_z$ tapes [9], appeared with a noticeably reduced intensity in Fig. 1(a) and 1(c). It indicates that the forma-tion and growth mechanism of Tl-1223 phase in the present composition may be different from that in $Tl_{0.8}Pb_{0.2}Bi_{0.2}Sr_{1.6}$-$Ba_{0.4}Ca_2Cu_3O_z$.

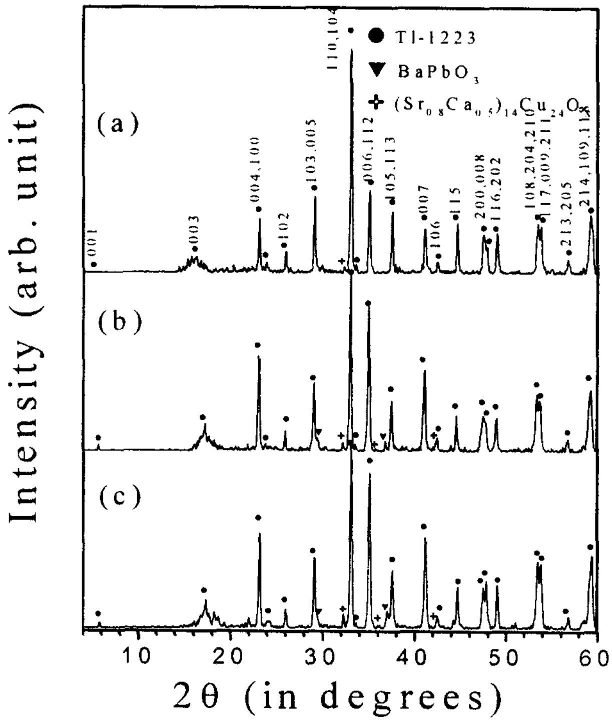
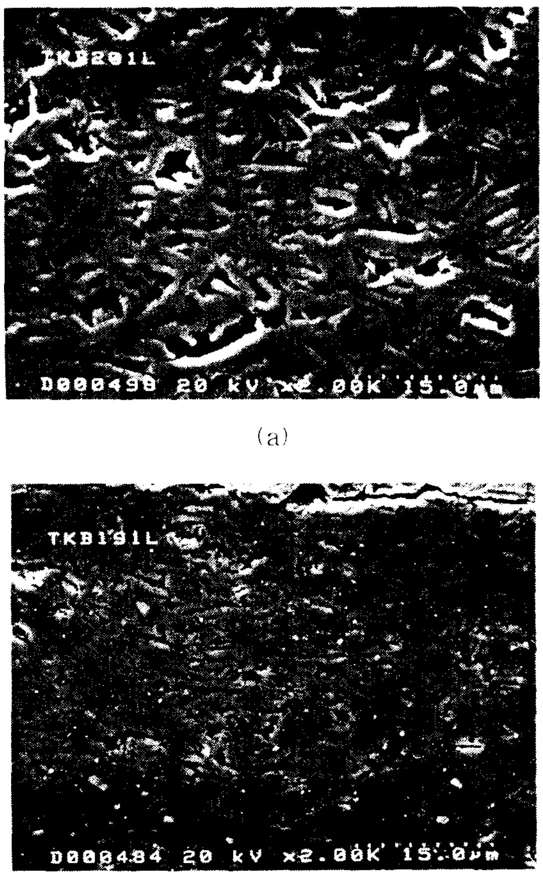


Fig. 1. XRD patterns taken in (a) a tape with J_c of 16,600 A/cm^2 heat-treated at 840℃ for 20 min, rolled and heat-treated at 830℃ for 7 h, (b) a tape with J_c of 20,900 A/cm^2 prepared by rolling the tape (a) and heat-treating again at 840℃ for 7 h, and (c) a tape with J_c of 25,200A/cm^2 heat-treated at 840℃ for 20 min, rolled, heat-treated at 840℃ for 4 h, rolled and heat-treated again at 840℃ for 4 h.

Fig. 2 shows SEM images taken in polished longitudinal surfaces of (a) a tape heat-treated at 840℃ for 20 min, rolled and heat-treated at 840℃ for 4 h, and (b) a tape with J_c of 22,300 A/cm^2 prepared by rolling the tape (a) and heat-treating at 840℃ for 3 h. In Fig. 2, plate-like Tl-1223 grains tend to be directionally aligned, at least in local regions, different from the cases of other compositions [3,6, 8,9]. It can be recognized that degrees of directional grain alignment and densification were much enhanced upon incorporating an intermediate rolling during the final HT. The visible enhancement of directional grain-alignment shown in Fig. 2(b) supports the enhanced intensities of (00l) peaks in Fig. 1(b). Fig. 2(b) also shows a possibility that J_c and its field dependence can be much enhanced provided that a proper TMT, which results in directional grain-alignment in a long range order, is found.



(a)

(b)

Fig. 2. SEM images taken in polished longitudinal surfaces of (a) a tape heat-treated at 840℃ for 20 min, rolled and heat-treated at 840℃ for 4 h, and (b) a tape with J_c of 22,300 A/cm^2 prepared by rolling the tape (a) and heat-treating at 840℃ for 3 h.

Fig. 3 shows the field dependence of magnetization J_c ($J_{c,m}$) calculated using Bean formula $J_{c,m}=20\Delta M/d$ from magnetic hysteresis loops measured in the tape with J_c of 24,900 A/cm^2 at selected temperatures. Here ΔM is magnitude of magnetization hysteresis and d average grain size, which is estimated to 7 μm. $J_{c,m}$ at 77 K and 1 T of the tape is estimated to 50,000 A/cm^2, which is higher by twice than that in the $Tl_{0.8}Pb_{0.2}Bi_{0.2}Sr_{1.6}Ba_{0.4}Ca_2Cu_3O_z$ tape [9].

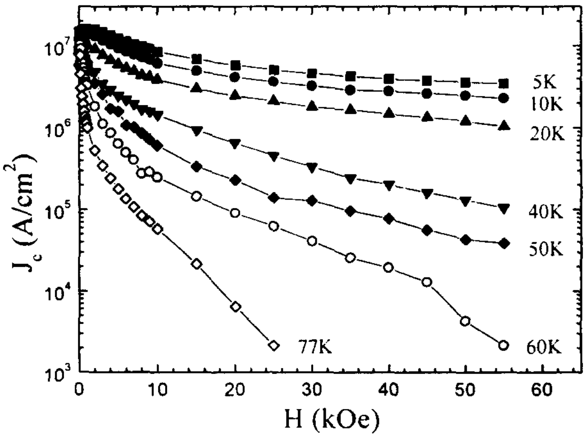


Fig. 3. Field dependence of magnetization J_c ($J_{c,m}$) calculated using Bean formula $J_{c,m}=20\Delta M/d$ from magnetic hysteresis loops measured in the tape with J_c of 24,900 A/cm^2.

Fig. 4 shows the field dependence of transport J_c measured at 77 K in a tape with J_c of 24,900 A/cm^2. The J_c starts to rapidly decrease near 50 Oe, and is saturated near 3,000 Oe. J_c at 77 K and 1 T is estimated to be 350 A/cm^2, which is lower by two orders of magnitude than the intragranular J_c ($J_{c,m}$). Such a great difference between inter- and intra-granular J_c's and the strong field dependence of J_c indicate that there exist significant weak-links in the tape, despite of such a high J_c at 77 K and 0 T, and the degree of directional grain-alignment observed is far from a desired one. Based on the report of Nabatame et al. [15], in which a great difference between inter- and intra-granular J_c's by two orders of magnitude started to appear in Tl-1223 thin film when the angles of tilt grain boundaries began to be greater than 10°, the transport property in the present tape seems to be dominated by large-angle grain boundaries. However, J_c of the present tapes start to drop at a little higher field and is saturated to a little higher value than that of $Tl_{0.8}Pb_{0.2}Bi_{0.2}Sr_{1.6}Ba_{0.4}Ca_2Cu_3O_z$ does [9]. This indicates that a little larger portion of intergranular contact survives as current paths in high fields in the present tapes than in $Tl_{0.8}Pb_{0.2}Bi_{0.2}Sr_{1.6}$-$Ba_{0.4}Ca_2Cu_3O_z$ tapes.

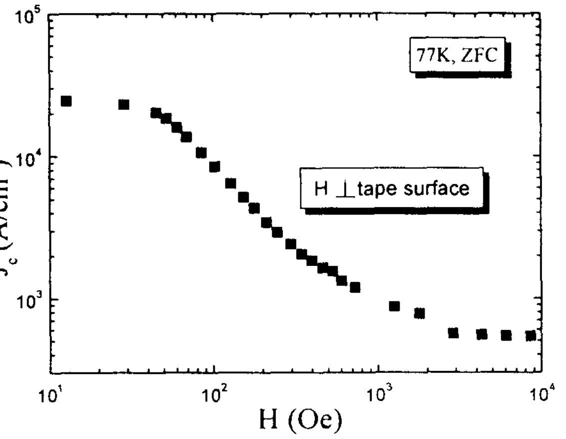
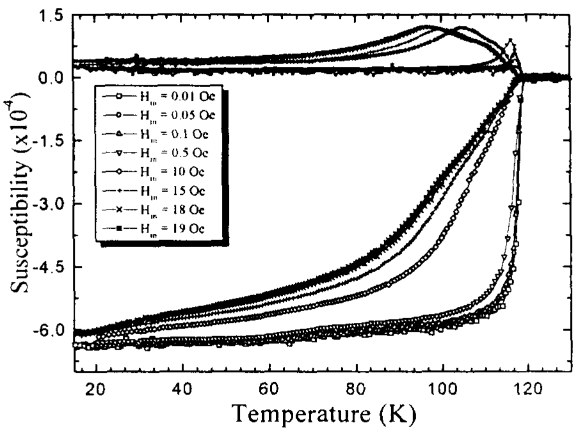


Fig. 4. Field dependence of J_c measured at 77 K in a tape with J_c of 24,900 A/cm^2.

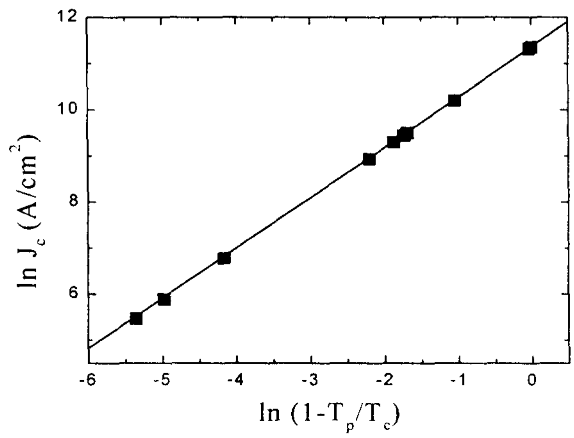
Fig. 5(a) shows the temperature depend-ence of real (χ') and imaginary (χ'') components of ac susceptibilities measured at ac magnetic fields (H_m) of 0.01-19 Oe in the tape with J_c of 24,900A/cm^2. In the framework of a "finite temperature" critical state model [16] for a granular superconductor, χ'' represents the intergra-nular bulk pinning loss. The temperature (T_p) in which maximum χ'' appears under a certain ac field means a critical state is established in the specimen.

Fig. 5(b) shows ($1-T_p/T_c$) dependence of J_c calculated by applying the critical state formula $J_c \approx H_m/a$ [17] to Fig. 5(a), where 2a is the superconducting core thickness of the tape and T_c the critical transition temperature, which is estimated to 119.0 K. It was plotted to evaluate characteristic of the inter-granular junction by estimating the β value in $J_c(T) \propto (1-T/T_c)^{\beta}$ [18]. Here As β is estimated to be 1.1 from Fig. 5(b), the inter-granular contacts seems to be mainly dominated by SIS junctions. Also J_c at 77 K and 0 T estimated from Fig. 5(b) is 26,200 A/cm^2, and is consistent with the measured transport J_c.

Tl-1223 grains in the present tapes are plate-like and directionally aligned, at least local regions, different from those in tapes of other compositions. Furthermore, incorporation of the intermediate rolling in the final HT process enhanced degrees of densification and directional grain-align-ment, and thus enhanced J_c's. By the way, such an J_c enhancement by incorpo-rating the intermediate rolling has not been observed in just-rolled Tl-1223 tapes. In the case of $Tl_{0.93}Bi_{0.22}Sr_{1.6}Ba_{0.4}Ca_2Cu_3O_z$ tapes, J_c could be enhanced by using an intermediate pressing, not by rolling [7]. In the case of $Tl_{0.8}Pb_{0.2}Bi_{0.2}Sr_{1.6}Ba_{0.4}Ca_2Cu_3$-$O_z$ tapes [9], J_c severely dropped upon incorporating the intermediate rolling during the final HT for 14h. The severe J_c decrease upon the intermediate rolling was ascribed to significant decomposition of Tl-1223 phase resulting from severe Tl evaporation during the relatively long HT [9]. In the present tapes heat-treated for around 14 h, however, the J_c value increas-ed by more than twice upon incorporating



(a)



(b)

Fig. 5. (a) Temperature dependence of ac susceptibilities measured at selected fields in the tape with J_c of 24,900A/cm^2 and (b) ($1-T_p/T_c$) dependence of J_c calculated by applying Fig. 5(a) to the critical state formula $J_c \approx H_m/a$.

the intermediate rolling, as shown in Table 1. The J_c enhancement by incorporat-ing the intermediate rolling are attributed to relatively facilitated grain-reconnection and microcrack heal-out during the final heat-treatment due to suppressed evapor-ation of Tl and excessive Ca content in the present composition. The possible ex-planations are as follows:

Incorporation of the intermediate rolling during the final heat-treatment enhances degree of densification in the superconduct-ing core, as well as directional grain-alignment in the present case. However, the intermediate rolling is also a process of grain fragmentation and microcrack generation. To get an excellent grain-con-nectivity and thus high J_c, the broken grains must be reconnected and the micro-cracks must be healed out during sub-sequent HT. By the way, it is thought that reconnection of the broken grains and healing out of the microcracks are accom-modated by formation and growth of new Tl-1223 phase from an unreacted mixture of Tl-1212 and (Ca,Cu)-rich liquid phase [19]. It is expected, however, that severe Tl evaporation during the subsequent HT causes already-made Tl-1223 phase to decompose, and new Tl-1223 to hardly form and grow by changing Tl content in the unreacted mixture, resulting in poor grain-connectivity and thus low J_c, as observed in $Tl_{0.8}Pb_{0.2}Bi_{0.2}Sr_{1.6}Ba_{0.4}Ca_2Cu_3O_z$ tapes [9]. However, in the present case where Tl evaporation is retarded, excellent grain-connectivity and thus high J_c can be achieved provided that formation and growth of new Tl-1223 phase participating in reconnection of broken grains is well controlled. Furthermore, as the present composition contains excessive Ca over the stoichiometric ratio of Tl-1223 phase, it will be beneficial to supply sufficient Ca necessary for the formation and growth of new Tl-1223, which is expected to slowly occur due to low mobility of Ca atoms. The reason why the intermediate pressing is effective in enhancing J_c and the inter-mediate rolling is not in other composi-tions seems to be as follows: It is well-known that rolling results in a higher degree of fragmentation and less densifi-cation and alignment than pressing does [19], due to its lower magnitude of pres-sure and more complicate stress distrib-ution. This means that rolling offers a higher density of channels for Tl evapora-tion and necessitates formation of larger amount of new Tl-1223 for grain-reconnec-tion and crack-healing during final HT. Therefore, it is expected that Tl-1223 grains after the intermediate rolling hardly reconnect each other, because a relatively small amount of unrected mixture is available at that stage and severe Tl evaporation results in a shortage of Tl necessary for formation of new Tl-1223 and even decompose already-formed Tl-1223. By the way, the reason why the decomposition of Tl-1223 phase is sup-pressed in the present composition is left to be unknown at present.

$\beta = 1.1$ of the present tape obtained from the ac susceptibility study appeared to be lower than that ($\beta = 1.37$ [20]) in $Tl_{0.8}Pb_{0.2}Bi_{0.2}Sr_{1.6}Ba_{0.4}Ca_2Cu_3O_z$ tapes, even though J_c value in the former tape is higher than that of the latter. This fact may mean that the latter tape have a larger fraction of SNS intergranular junc-tion than the former, and thus the J_c value in Tl-1223 tapes depends on grain contact areas rather than intergranular bonding nature.

4. Conclusion

In the present study, the grain morpho-logy, the changes in morphology and J_c with the TMT history, the field depend-ence of J_c and the nature of intergranular bonding were studied in Tl-1223/Ag tapes of $Tl_{0.8}Pb_{0.2}Bi_{0.2}Sr_{1.8}Ba_{0.2}Ca_{2.2}Cu_3O_z$ nominal composition prepared using the powder-in-tube method incorporating an in-situ reaction method. The tape core showed the morphology in which plate-like Tl-1223 grains tend to be aligned in preferred orientations, at least in local regions. Furthermore, grain-connectivity and direc-tional alignment of the grains were much enhanced by incorporating an intermediate rolling during the final heat-treatment process. As a result, high J_c values near 2.5×10^4 A/cm^2 at 77 K and 0 T with an excellent reproducibility were obtained in just rolled tapes. The strong field depend-ence of J_c even in low fields, however, indicated that there still existed signifi-cant weak-links and the degree of direct-ional grain-alignment was far from the desired one. The intergranular binding in the tapes seemed to be mainly dominated by a SIS junctional type. The high J_c's seem to result from relatively easy recovery of excellent grain-connectivity during the final HT after the intermediate rolling, probably due to retarded Tl evaporation and excessive Ca content in the present composition, and are attri-buted to enhanced contact areas between Tl-1223 grains rather than enhanced directional grain-alignment. The present study shows a possibility that J_c and its field dependence can be enhanced provided that proper TMT, which results in directional grain-alignment in a long range order, is found.

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