

**EFFECT OF INTERMEDIATE ROLLING ON THE TRANSPORT
PROPERTIES OF $(\text{Bi}_{1.6}\text{Pb}_{0.4})\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ SUPERCONDUCTOR
TAPES WITH NANO NiFe_2O_4 ADDITION**

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ABSTRACT

The effects of nano sized NiFe_2O_4 addition in $(\text{Bi}_{1.6}\text{Pb}_{0.4})\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ (Bi2223) superconductor tapes were investigated. The $(\text{Bi}_{1.6}\text{Pb}_{0.4})\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ powders were prepared by using coprecipitation technique and the tapes were prepared by using the powder-in-tube method (PIT). 0.01 wt% nano sized NiFe_2O_4 (13 nm) was added to the powders as this ratio of nano NiFe_2O_4 optimized the critical current density (J_c) of bulk samples based on our initial studies. The effects of intermediate rolling on the transport critical current density of the $(\text{Bi}_{1.6}\text{Pb}_{0.4})\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ - $(\text{NiFe}_2\text{O}_4)_{0.01}$ tapes were investigated. The transport critical current density at 30 K was 17870 A/cm² for tapes sintered for 50 hours without intermediate rolling. Intermediate rolling up to two times increased the J_c at 30 K to 32700 A/cm². Intermediate rolling improved the J_c of the tapes by 83 % compared to tapes without intermediate rolling.

INTRODUCTION

The transport critical current density of high temperature superconductor tapes is one of the interests in superconductor research today. Several methods have been used to improve the J_c of the tapes. One of the effective methods is employing artificial impurities as pinning centers to the flux line network [1-3]. Magnetic impurities such as $\gamma\text{-Fe}_2\text{O}_3$ and Fe_3O_4 were added to Bi2223 tapes to perform as flux pinning centers [4-5]. Ag-sheathed Bi2223 tapes fabricated by powder-in-tube method have been widely studied. Many efforts have been done to investigate the conditions to improve J_c of Bi2223/Ag tapes. Several studies have proven that an optimum combination of heat treatment and intermediate mechanical rolling can effectively improve J_c of Bi2223/Ag tapes [6-7]. The mechanical treatment was found to improve the alignment and connectivity of the grains in Bi2223 tapes [8-9]. In this paper, we report the experimental results of the flux pinning behavior of Bi2223 tapes with different sintering period and intermediate mechanical rolling. The effect of intermediate rolling on the transport critical current density is also presented.

EXPERIMENTAL METHOD

The solid state reaction and precursor method have been employed to prepare polycrystalline samples with nominal starting composition $\text{TlSr}_2(\text{Ca}_{1-x}\text{Yb}_x)\text{Cu}_2\text{O}_7$ with $x = 0.1, 0.3, 0.4, 0.5$ and 0.6 . High purity oxides ($\geq 99.99\%$) of SrO , CaO , Yb_2O_3 and CuO were mixed in appropriate amounts and heated at 900°C for over 24 hours with several intermittent grindings. The heated precursor were then mixed completely with appropriate amounts of Tl_2O_3 and then pressed into pellets of 13 mm diameter and 3 mm thickness. The pellets were heated at 1000°C in flowing O_2 followed by oven cooling.

The samples were characterized by powder X-ray diffraction (XRD) method using a Siemens D 5000 diffractometer with Cu-K_α source. The volume fraction of 1212 and 1201 phase were estimated by assuming that the amounts of 1212 and 1201 phases are proportional to the strongest diffraction line of each phase, i.e. the (103) reflection of the 1212 phase and the (102) reflection of the 1201 phase. The lattice parameters were calculated by employing at least 10 diffraction peaks. The electrical resistance (dc) were measured with the 4-point probe method. The scanning electron micrographs were recorded using a Philips XL 30 Scanning Electron Microscope (SEM).

EXPERIMENT DETAILS

$\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ superconductor powders were prepared by using the co-precipitation technique. 0.01 wt% nano sized NiFe_2O_4 was added to the ultra-fine powders. This ratio is chosen based on our initial studies on bulk samples where 0.01 wt% nano NiFe_2O_4 optimized the J_c [10]. The mixture of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ powders and nanosized NiFe_2O_4 were ground and packed into Ag tube. The tube was rolled and swaged into wire. The wire was cut into 2 cm long sections and rolled into tape form. The tapes were divided into five groups. Each group of tapes was sintered at 845°C for different sintering period and intermediate rolling as summarized in Table 1. Each group was labeled as N5, N10, N15, N55 and N555 respectively. Tape without addition of NiFe_2O_4 was also prepared as comparison. The critical current was measured using the four-point probe method in conjunction with CTI cryogenics closed-cycle refrigerator (Model 22). The transport critical current density (J_c) was determined at 30 K and 77 K by using the $1\ \mu\text{V}/\text{cm}$ criterion. The transport critical current density was calculated by using the equation $J_c = I_c/A$, with I_c is the critical current and A is the cross sectional area. The phases in the samples were identified using the powder X-ray diffraction (XRD) method by using a Siemens D 500 diffractometer with CuK_α radiation. The microstructure was observed by a Scanning Electron Microscope (SEM). A Philips energy dispersive X-ray (EDX) analyser PV99 was used to observe the distribution of nanosized NiFe_2O_4 in the tapes.

RESULTS AND DISCUSSION

Table 1 shows the transport critical current density (J_c) at 30 K and 77 K, and volume fraction of 2223:2212 phases for all the tapes. Volume fraction of Bi2223 phase and Bi2212 phase were calculated. All samples showed a dominant phase of Bi2223 with a minor phase of Bi2212. However, longer sintering period with intermediate rolling degraded the Bi2223 phase formation.

Table 1: Details of sintering period and intermediate rolling treatment for all tapes. Transport critical current density (J_c) at 30 K and 77 K, and volume fraction of 2223:2212 phase for all the tapes.

Sample	Heat Treatment	Number of intermediate rolling	J_c at 30 K (A/cm ²)	J_c at 77 K (A/cm ²)	Volume fraction 2223:2212 (%)
Pure	50	0	12400	3090	72:28
N5	50	0	17870	3970	76:24
N10	100	0	21600	4630	67:33
N15	150	0	22230	6730	67:33
N55	50+50	1	24900	6950	71:29
N555	50+50+50	2	32700	7100	65:35

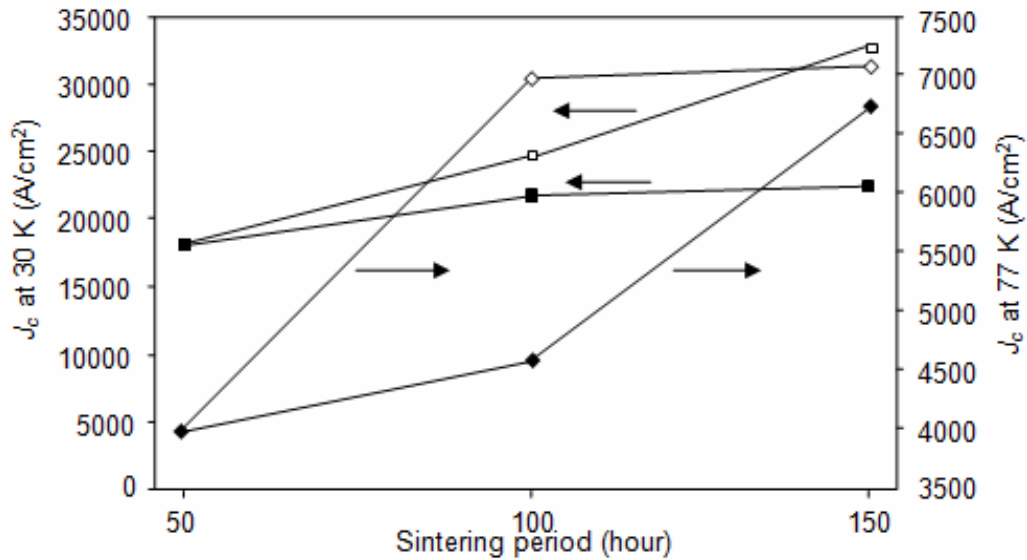
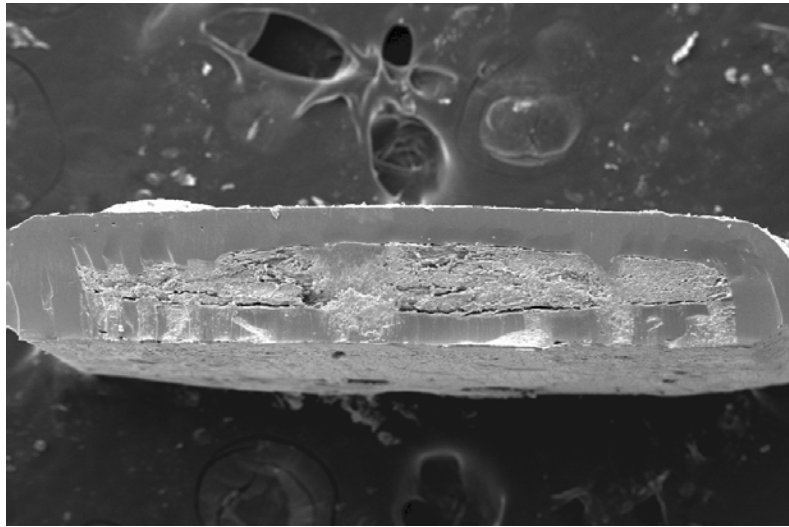


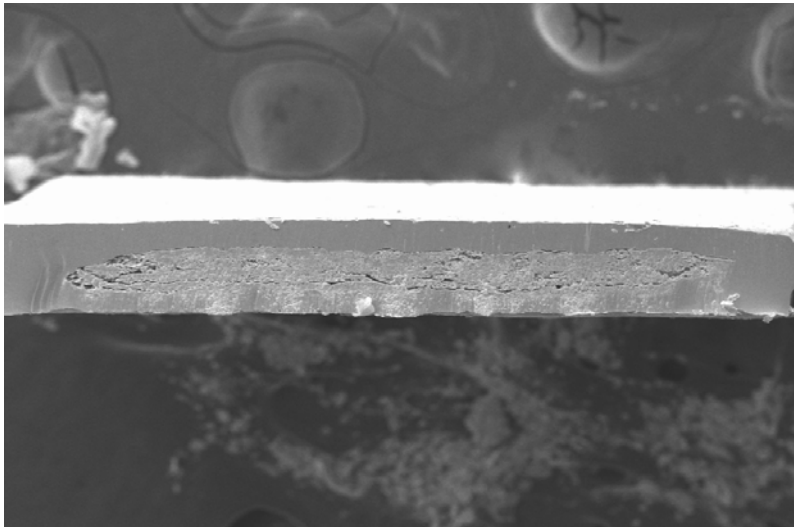
Figure 1: Transport critical current density (J_c) at 30 K and 77 K versus sintering period for tapes with and without intermediate rolling.

Figure 1 shows the curves of transport critical current density (J_c) at 30 K and 77 K versus sintering period for tapes with and without intermediate rolling. 0.01 weight %

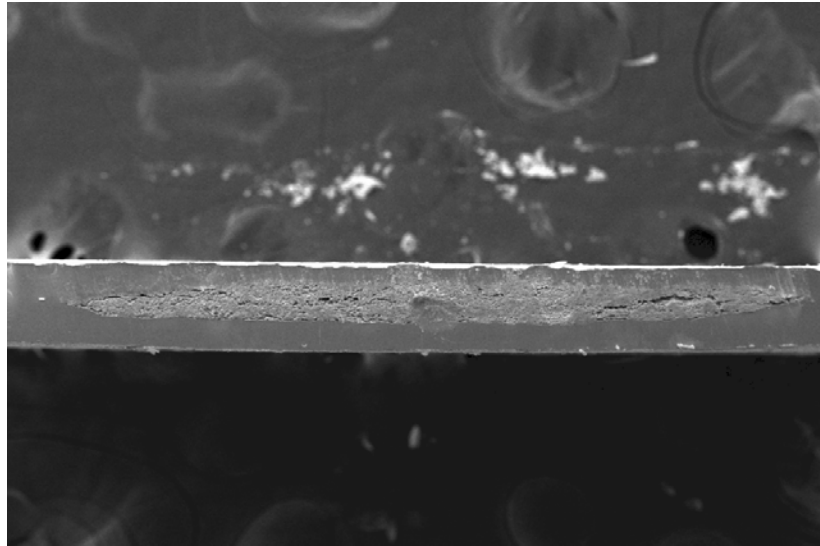
nano NiFe_2O_4 was introduced into Ag-sheathed $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ (Bi2223) tapes to perform as pinning centers in order to enhance the J_c of the tapes. At 30 K and 77 K, the NiFe_2O_4 added tape showed higher J_c compared to the tape without nano NiFe_2O_4 addition for sintering period of 50h. The superconductor tapes were subjected to different sintering period and intermediate mechanical rolling. When sintering period was prolonged to 150h, the J_c of tapes with and without intermediate rolling was enhanced. However, for sintering period of 150h, the tape with intermediate rolling exhibited J_c of 32700 A/cm^2 , which is higher than 22230 A/cm^2 of tape without intermediate rolling.



(a)



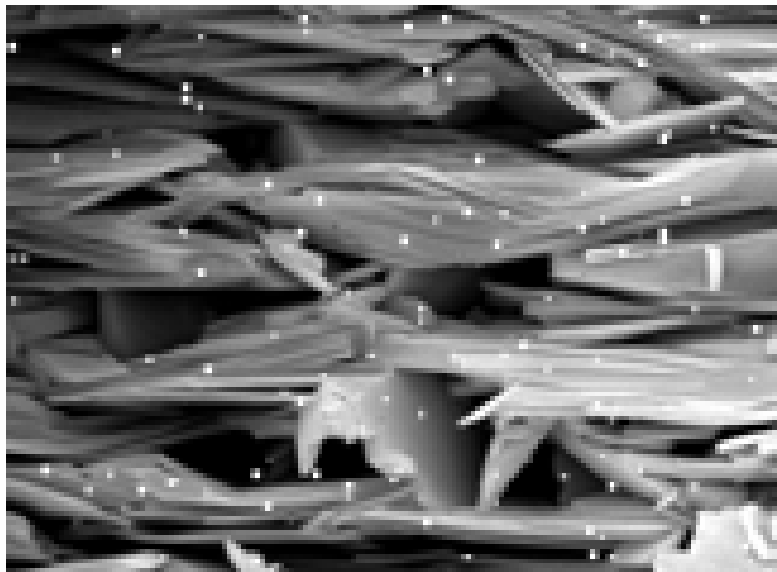
(b)



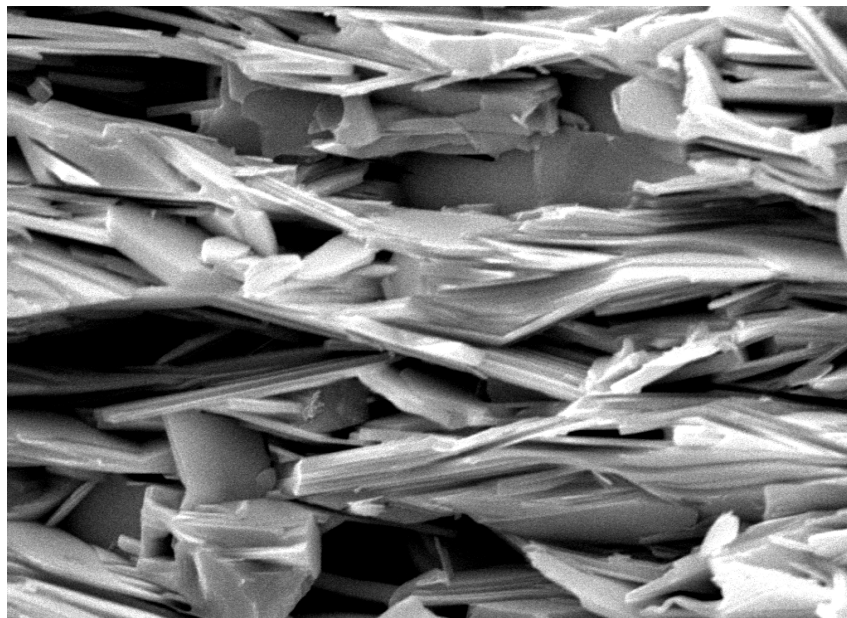
(c)

Figure 2: SEM micrographs on the cross section of tapes N5, N55 and N555.

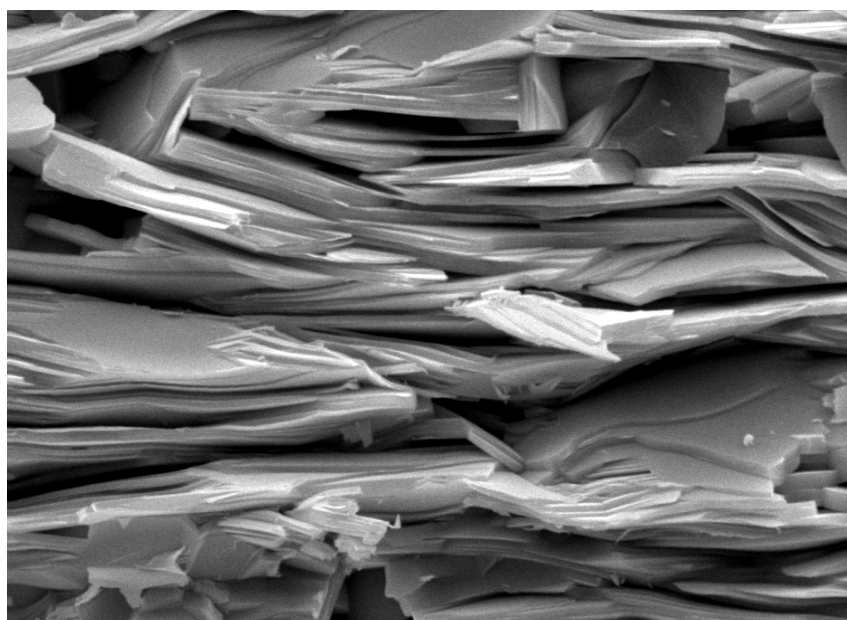
Figure 2(a), (b) and (c) show the SEM micrographs on the cross section of tapes N5, N55 and N555. Tapes N55 and N555 are the tapes with 1 and 2 times of intermediate rolling, respectively. The increase of intermediate rolling on the tapes has decreased the cross sectional area of the core. Therefore, the increase in J_c of tapes with intermediate rolling is mainly due to the decrease in the cross sectional area of the tapes.



(a)



(b)



(c)

Figure 3: SEM micrographs of tapes N5, N15 and N555.

Figure 3(a), (b) and (c) show the SEM micrographs of tapes N5, N15 and N555. The

white dots in Figure 4(a) indicated the distribution of nano NiFe_2O_4 . The mechanical treatment was found to improve the alignment and connectivity of the grains in these Bi2223 tapes. This is also one of the reasons that can explain the increase of J_c of tapes with intermediate rolling.

CONCLUSION

Nano NiFe_2O_4 (0.01 wt%) showed effective flux pinning capability which enhanced the transport critical current density of tapes $(\text{Bi}_{1.6}\text{Pb}_{0.4})\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}-(\text{NiFe}_2\text{O}_4)_{0.01}$. Thermo mechanical treatment has played an important role in enhancing the transport properties of tapes. Intermediate rolling improved the J_c of the tapes by 83 % compared to tapes without intermediate rolling. The enhancement of transport critical current density in tapes with intermediate rolling is due to the decrease in cross sectional area of the core and the improvement of grain alignment and connectivity.

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