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AUTHOR(S): ~~J. O. Willis, STC, M. P. Maley, STC, P. J. Kung, STC, J. Y. Coulter, STC, D. E. Peterson, STC,~~ P. G. Wahlbeck, Wichita State University, J. F. Binger MST-6, D. S. Phillips, MST-4

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TRANSPORT AND MAGNETIZATION CRITICAL CURRENT DENSITIES IN $TlBa_2Ca_2Cu_3O_x$ Tapes†

J. O. Willis, M. P. Maley, P. J. Kung, J. Y. Coulter, D. E. Peterson, P. G. Wahlbeck*, J. F. Bingert, and D. S. Phillips
Superconductivity Technology Center, MS-K763, Los Alamos National Laboratory, Los Alamos, NM 87545 USA

Abstract--The powder in tube process was used to produce silver-sheathed tapes of $TlBa_2Ca_2Cu_3O_{8+x}$ (Tl-1223). The powder was produced by thalliating a precursor powder mixture to produce the Tl-2223 phase and then heating to drive off excess Tl and reach the Tl-1223 stoichiometry. The tapes were rolled and pressed, each step followed with a 3 h sintering. The 200 μm thick tapes show little sign of texturing; however, the critical current shows a small (~50%) dependence on the direction of the applied magnetic field. Both transport and magnetization measurents indicate relatively strong pinning at high temperatures. The 75 K self field critical current density is 62 MA/m^2. Transport measurements reveal the presence of weak links at all temperatures, but with a relatively weak field dependence above ≈0.1 T.

I. INTRODUCTION

The development of conductors from high T_c superconductors (HTS) is controlled by many properties, of both materials (i. e. processing, phase equilibria) and fundamental (intrinsic flux pinning, electronic structure, etc.) origin. Each of the HTS materials considered for applications, $YBa_2Cu_3O_x$ (Y-123); the Bi-based compounds $Bi_2Sr_2CaCu_2O_x$ (Bi-2212) and $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ (Bi-2223); and the Tl-based (Tl,Pb)-(Ba,Sr)-Ca-Cu-O (representative of which Tl-2223, Tl-1223, and Tl-1212 will be discussed here) has both good and bad features. The double Bi (or Tl) planes separate the Cu-O planes thought to be responsible for superconductivity in both Bi systems and in the double layer Tl systems. This large separation may partially decouple the superconducting volumes along the c-axis direction resulting in "pancake" magnetic vortices and materials that show poor high-temperature, high-field properties.[1] Materials with single "insulating" layers such as Y-123 and Tl-1223 are known to have better properties in this regime.[2] We report on Tl-1223 powder in tube tapes[3] that show promising high temperature characteristics.

II. EXPERIMENTAL

The $TlBa_2Ca_2Cu_3O_{8+x}$ powder was synthesized by first producing the precursors $BaCuO_2$ and Ca_2CuO_3 from carbonates and CuO and then mixing these with Tl_2O_3 powder, forming a pellet and sintering in an oxygen environment (PO_2 ~ 1600 Torr) using a covered gold boat in a quartz tube for 3 h at 925°C to produce the Tl-2223 phase. In a series of heating stages near 900°C (at PO_2 ~ 830 Torr) with intermediate grindings, sufficient thallium was volatilized and carried away in the flowing oxygen gas to produce the desired Tl-1223 phase. The powder was characterized by x-ray diffraction, microscopy, and ac and dc magnetic susceptibility.

The powder was ground to a median grain size of 18.6 μm before filling the 6.35 mm OD by 4.35 mm ID by 100 mm long Ag tube, which was then drawn to 1 mm diam and rolled to a thickness of 240 μm. Following DTA studies of the as rolled tape, it was determined that no reaction occured with the silver for temperatures below about 840°C. After further investigation of processing parameters, the tape was sintered at 775°C for 3 hours in flowing O_2, pressed at 1.2 GPa to a final dimension of 200 μm thick by ~3 mm wide and then resintered under the same conditions as before. The microstructure was analyzed by optical microscopy. The superconducting properties of the tape were determined by SQUID and ac magnetometry and by transport critical current density J_c measurements as a function of temperature and magnetic field using a 0.5 μV/cm criterion.

III. RESULTS

The Tl-1223 phase is produced after the intermediate formation of the Tl-2212 and Tl-2223 phases and the subsequent loss of thallium during the sinterings. The powder consists of nearly single phase (by x-ray diffraction) agglomerates of very small Tl-1223 platelets with a small amount of $BaCuO_2$ impurity. The superconducting transition temperature T_c of the powder in a 1 mT field was ~110 K with a small tail up to ~125 K indicating the presence of some Tl-2223 phase. In the tape, the platelets were not observed to texture nor did they show as much grain growth as is seen in Bi-2223 tapes. This is probably because of the less graphitic, more three-dimensional nature of the Tl-1223 crystal structure and the much shorter processing time in the tape (3 h vs ~100+ h for Bi-2223), respectively. Most small cracks were found to heal after the first sintering step but larger ones remain. The void volume produced by the first sintering is partially decreased by the pressing step thus improving connectivity (and the J_c) of the core.

Both rolled/sintered and second step pressed/sintered tapes show a dc susceptibility onset T_c at 110 K, with a broader width than for the powder. The slighly sharper trasnsition for the pressed tape relative to the rolled indicates possible greater healing of the damage from the mechanical processing. T_c and transition widths from ac susceptibility of both powder and pressed tape are in good agreement with the dc measurements. The very broad loss peak in χ'' shifts about

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*Permanent Address: Chemistry Dept., Wichita State Univ., Wichita KS 67208

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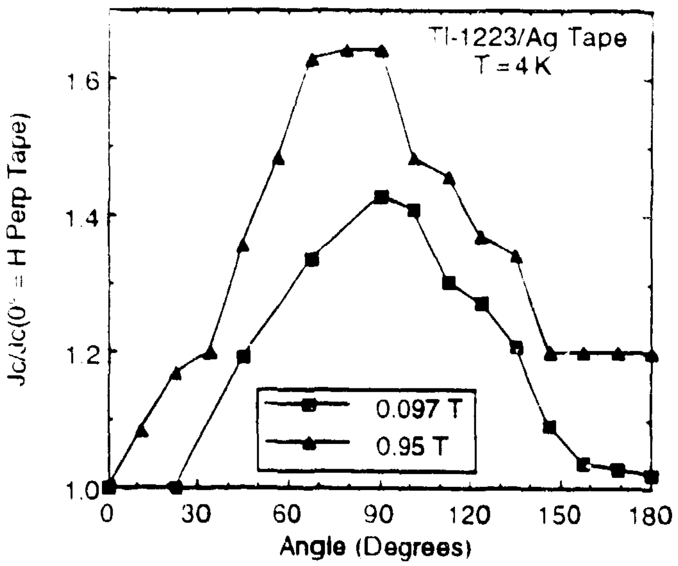

Fig. 1 Normalized transport critical current density J_c at 4 K vs. angle for magnetic fields below and above the weak link region. The J_c values at 0°(H ‖ c) are 49 MA/m^2 and 4.2 MA/m^2 at 0.097 T and 0.95 T, respectively.

10 K at f = 500 Hz and $\mu_0 H$ = 1 mT when the dc field is increased from 0 to 20 mT. The dc magnetic hysteresis loop at 35 K remains open to much higher fields than for Bi-2223 (> 5 T vs. 3 T), indicating that irreversible behavior ($J_c \neq 0$) extends over a much larger temperature-magnetic field region. Thus the phase space for practical usage of conductors based on the Tl-1223 system may be expected to extend to higher temperatures than for Bi-2212 or Bi-2223 based tapes.

Transport J_c results yield a value of 62 MA/m^2 at 75 K and self field for the pressed tape. This value decreases quickly at fields below ~0.1 T, indicating the presence of weak links. However, the field dependence above the low field region (Fig. 1) is much weaker than for Bi-2223 at the same temperature and indicates the potential application advantages of Tl-1223.

The current-voltage characteristics at 4 K and 75 K of a tape sample have been fitted to a power law ($[E/E_0] = c\,[J/J_0]^n$, where E is the electric field, J is the current density, and E_0, J_0, and c are numerical constants) in the region ~10^{-9} to 10^{-7} V/m: above this field range, the characteristic departs from a power law and some of the current may be shunted by the silver sheath. The results of the fits are shown in Fig. 2; the sharp drop in the n value occurs in the same field region as the sharp drop in J_c associated with the break down of the weak links. The relatively constant n value with field above this region, especially for the 75 K data, again indicates the connectivity of the remaining current paths as well as the presence of strong intragranular pinning. For a roughly comparable quality Bi-2223/Ag tape (75 K self-field J_c of 160 MA/m^2) [7], n values decline much more rapidly, dropping below 2 at 64 K and 1 T (H ⊥ c); here the n value is limited not by intergranular weak links, but by the weak intrinsic pinning within the grains because of the very small activation energy needed to overcome the pinning potential of the loosely coupled pancake vortices.

The weak links were also apparent by comparing the magnetic hysteresis loops for the ground powder, with which the silver tube had been packed, with that of the tape; the magnetization did not scale with the tape size, indicating that there were only small intergranular currents because of the weak links and that the true size of the magnetization current loops was the grain size. Although no texture was evident from the metallographic examination of the tape sections, there was evidence of grain alignment in the dependence of J_c on the orientation of the magnetic field to the tape plane. The 4 K angular anisotropy (≈ 50%, both in and above the weak-link region), with the larger value for the field in the tape plane, clearly indicates this as shown in Fig. 3. Anisotropy was also observed at 75 K.

IV. DISCUSSION

The lack of significant texturing in these Tl-1223 tapes leads to high-angle grain boundaries and weak-link behavior very similar to that seen in Y-123. Other workers have reported similar results on Tl-1223 and Tl-1212 based powder in tube process tapes.[3, 5] Efforts to improve grain alignment may be achieved by several approaches. Preliminary experiments with Tl/Bi-1212 powders have produced particles with a platy, high aspect ratio morphology [8] compared to the Tl-1223 powders; this particle shape should be more beneficial to deformation induced texturing. Rolling the tapes to a smaller initial thickness, not possible in the present experiment because of particle size limitations, may also result in increased texture. Finally, increased grain growth, achieved by either sintering for longer times or at

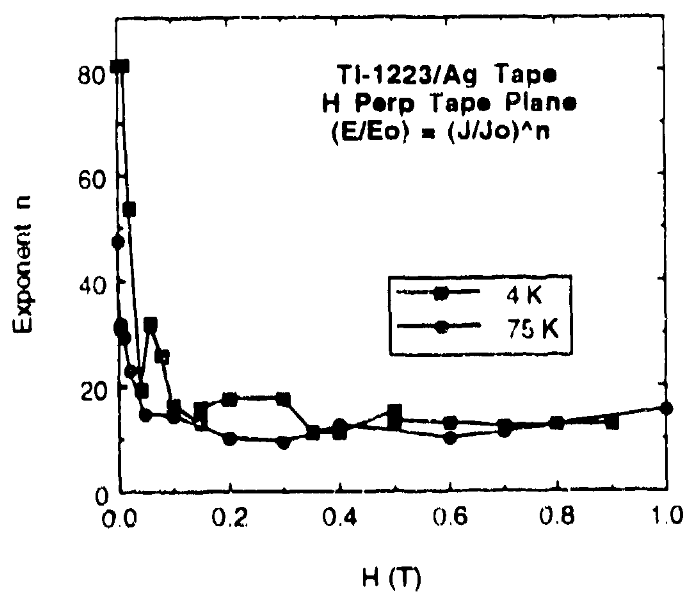

Fig. 2 Power law exponent n of the current density J - electric field E characteristic vs. magnetic field H applied perpendicular to the tape plane for a pressed Tl-1223 tape.

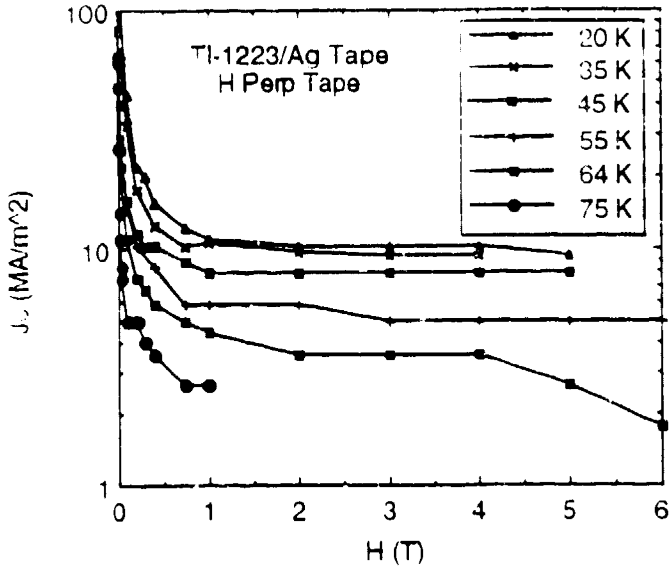

Fig. 3 Transport critical current density J_c at selected temperatures vs. magnetic field $\mu_0 H$ applied perpendicular to the tape plane for a pressed Tl-1223 tape.

higher temperatures or both, is expected to result in larger aspect grains and better alignment. All of these subjects are currently under investigation.

The upper limit of the achievable J_c in a tape can be approximated by that of high quality thin films. Recently, polycrystalline spray pyrolized films of Tl-1223 on YSZ have been reported to reach 1000 MA/m^2 at 77 K and self field and 100 MA/m^2 at 65 K and 2 T.[9] These 2-3 μm thick films are not single crystalline but do show a high degree of c-axis texturing. Thus such J_c values should also be possible in a long conductor if a similar level of grain alignment can be achieved.

V. CONCLUSIONS

Powder in tube process tapes of Tl-1223 using a silver sheath have been prepared from solid state reaction precursor powders which were then thalliated. The best results were achieved on tapes rolled/sintered and pressed/sintered using a 775°C, 3 h heat treatment after each mechanical process. The 200 μm thick tapes show good high field properties (i.e., relatively flat J_c-H curve, I-V power law n values of 10) even at high temperatures. These properties reflect the relatively large pinning energy of these single insulating layer superconductors. However, more work is needed to improve the texturing through improvements in powder morphology and thermomechanical processing, and to reduce the resultant weak-link behavior, apparent in the rapid drop of J_c in fields less than ~0.1 T, and to further increase pinning at the higher temperatures.

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