# The Effect of the Maximum Processing Temperature on the Microstructure and Electrical Properties of Melt Processed Ag-sheathed $Bi_2Sr_2CaCu_2O_x$ Tape

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**Abstract—The critical current density ($J_c$) is very sensitive to the maximum temperature ($T_m$) used to melt process Ag-sheathed $Bi_2Sr_2CaCu_2O_x$ tapes. In this study we have found that the optimum $T_m$ was 894-896°C and that variations of ±2°C strongly decreased $J_c$. We found that the density of the oxide core and its Vickers hardness displayed a maximum in tapes that had been processed at the optimum $T_m$. In addition, the room temperature electrical resistivity of the core was lowest for the tapes with the maximum $J_c$ at 4.2 K. A formation of macropores was observed in tapes processed at $T_m$ above the optimum one. MO imaging and SEM observation showed that the magnetic flux penetrates more easily into tape areas containing macropores. Together these observations show that $T_m$ exerts a very powerful effect on the macroscopic density of the oxide core, this controlling the connectivity and the effective cross-section of the oxide core. Thus $J_c$ depends on $T_m$ primarily because the effective cross-section of the core depends on $T_m$.**

## I. INTRODUCTION

Melt processing is used to prepare Ag-sheathed $Bi_2Sr_2CaCu_2O_x$ (2212) tape with high overall critical current density ($J_c$) [1,2]. The composition and characteristics of the powder, the mechanical processing of the tape, and the heat treatment all affect $J_c$, where $J_c$ is defined by the critical current $I_c$ divided by the total Bi-Sr-Ca-Cu-O (BSCCO) core cross-section. Recently it has been shown that there is a strong dependence of $J_c$ (4.2K) on $T_m$, the maximum temperature used for melt processing [3,4]. The present work examines how and why $J_c$ varies with $T_m$, and correlates $J_c$ and $T_m$ with physical properties of the 2212 core. We melt processed samples under identical conditions, varying only $T_m$, then measured the critical current ($I_c$) and the average cross sectional-area (A) of the tape and calculated $J_c$. Polished transverse and longitudinal cross sections were examined using SEM in order to observe porosity, non-superconducting grains, and the 2212 alignment. We also measured the density of the core, its hardness, which in 2223 tapes was found to correlate with $J_c$ [5], the room temperature electrical resistivity of the core, and visualized magnetic field penetration into the BSCCO core using magnetooptical imaging.

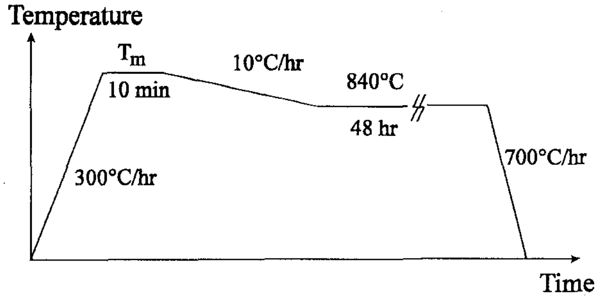

Fig. 1 Melt processing schedule used in this experiment. The samples were processed in pure $O_2$ at 1 atm.

## II. SAMPLE PREPARATION

The samples were prepared from a single core tape that contained powder with a nominal composition of $Bi_{2.1}Sr_2CaCu_{1.95}O_x$. The outer tape dimensions were 2.95 mm x 0.158 mm; the cross sectional area of the core was $1.3x10^{-3}$ $cm^2$. Figure 1 shows the melt processing schedule, which was done in 1 atm of pure $O_2$. $T_m$ was varied from 892°C to 900°C. To reduce the number of processing cycles needed to cover this temperature range, we melt processed tapes that were 125 mm to 150 mm long, which is longer than the uniform temperature zone in the processing furnace (Fig. 2). Thus the ends of each tape were processed at lower temperatures than the central tape section. The processed tapes were cut into 3 or 4 sections and 2 current leads and six potential taps (~5mm apart from each other) were soldered onto the tape. V-I curves were measured at 4.2K and $I_c$ corresponding to 1 µV/cm between each set of taps was determined from the V-I curve. This allowed us to obtain the "local" $I_c$ values along the tape and to correlate $I_c$ to $T_m$. To test whether it was $T_m$ or the gradient in temperature that influenced $I_c$, we processed samples having two different positions in the furnace, as shown in Fig. 2b and 2c. Sections of the tapes processed as shown in Fig. 2b

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and the samples processed as shown in Fig. 2c with the same local $T_m$ had very similar $I_c$ values. This indicated that it was $T_m$ and not the gradient in temperature that affected $I_c$.

## III. RESULTS AND DISCUSSION

### *A. Critical Current Density Measurements*

Figure 3 shows the $J_c$ in the superconducting core measured at 4.2 K and $B_e$=0 as a function of $T_m$ for all the samples. The optimum $T_m$ is between 894°C and 896°C; the corresponding maximum $J_c$ is ~$2.6x10^5$ A/cm$^2$. There is a sharp maximum in $J_c$ with $T_m$. A deviation of ±2°C from $T_m$ reduces $J_c$ by about 25%.

### *B. Scanning Electron Microscopy Observations of Longitudinal and Transverse Tape Sections*

Sections of tape were mounted and polished to show transverse and longitudinal cross sections. Energy dispersive spectroscopy showed that the non-superconducting grains are the same in all the samples, consistent with earlier studies [6]. In samples processed at higher $T_m$, the volume fraction of macroscopic pores reducing the core cross-section and disturbing the 2212 grain alignment increased.

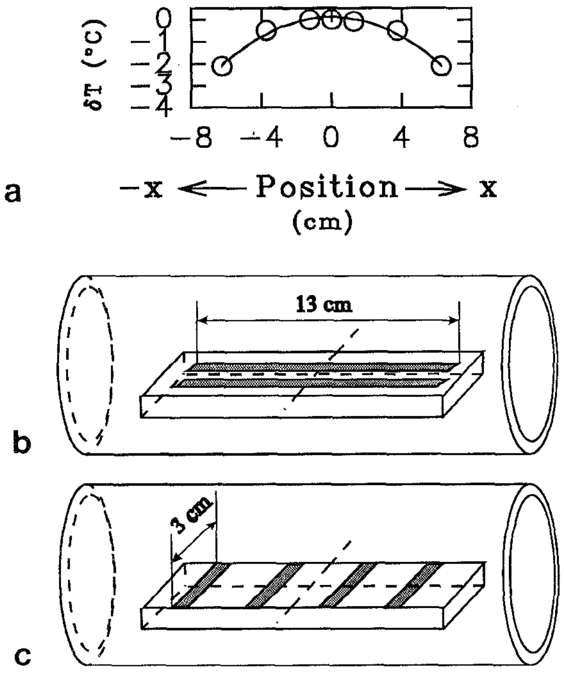

Fig. 2 (a) The temperature profile along the furnace axis. (b) "Longitudinal" samples where the tapes are longer than the constant temperature hot zone. (c) "Transverse" samples where the temperature is constant along the length of each sample.

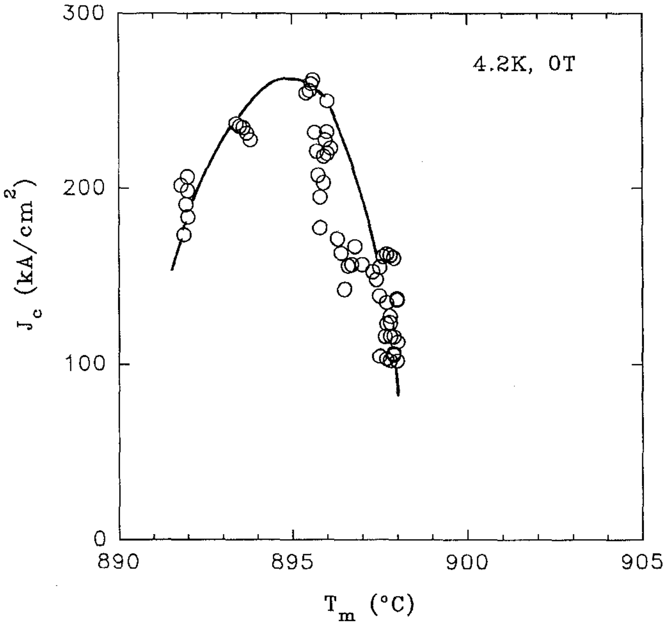

Fig. 3 The critical current density $J_c$ in the core at 4.2K and B=0 as a function of $T_m$.

### *C. Density Measurements*

The density of the 2212 core was measured using Archimedes' principle. The Ag sheath was removed chemically using an $HNO_3$-$H_2O_2$ etch from 8 mm long samples of fully processed tape and the mass of the core measured in air and in ethyl alcohol. Figure 4 shows the density of the samples as a function of processing temperature. The sample processed close to the optimum $T_m$ has the maximum density of 5.7 g/cm$^3$, as compared to a theoretical value of 6.5 g/cm$^3$. Samples processed at lower and higher $T_m$ had slightly lower density. The decrease in density for samples with $T_m$ higher than the optimum appears to be due to the increased formation of macro pores. Figure 4 also shows there is a direct correlation between the density and $J_c$.

### *D. Microhardness*

The hardness was measured with a Vicker's microindenter at 13 points on the longitudinal and transverse sections of each tape. Figure 5 shows the mean value and standard deviation of the hardness as a function of $T_m$. Even though there is significant scatter in the data, the data show that the sample processed at $T_m$ that gave the highest $J_c$ also had the maximum hardness. This finding is consistent with the density measurements, as the microhardness is thought to be related to the density, increasing with increasing density [5].

### *E. Room Temperature Resistivity Measurements of the BSCCO Core*

We chemically removed the Ag sheath from the tape leaving a small amount of Ag on each end of the tape for current leads and two islands of Ag between the ends of the tape for potential leads. Figure 6 shows the room temperature resistivity for the oxide core plotted against the tape $J_c$ values for tapes processed at different $T_m$. These data are strongly correlated, indicating that the "effective" cross-section of tapes and/or the ab-plane grain alignment are affected by $T_m$. Also shown in this figure is the range of room temperature resistivity reported for 2212 single crystals ($0.3\text{-}0.5\times10^{-5}$ $\Omega{\cdot}m$). We see that the room temperature resistivity of samples with high $J_c$ is twice as high as that of single crystals.

### *F. Magneto-optical Imaging of Flux Penetration into the BSCCO Core*

We used magneto-optical (MO) imaging to observe the penetration of external magnetic field into the tapes. MO images are sensitive to the perpendicular component of the magnetic field as detected in the plane of the indicator film, which is placed a few μm above the surface of the tape. The samples were cooled to 15 K in zero field, 20 mT was then applied perpendicular to the broad face of the tape. Figure 7 shows MO images taken several seconds after the field was applied. The magnetic field penetration of all samples was quite inhomogeneous. Fig. 7 shows that the sample processed at the optimum temperature $T_m$ was penetrated by less flux than the other samples. The inhomogeneity of the field penetration in 2212 tapes is very different from the more uniform penetration of the magnetic field into 2223 tapes [7].

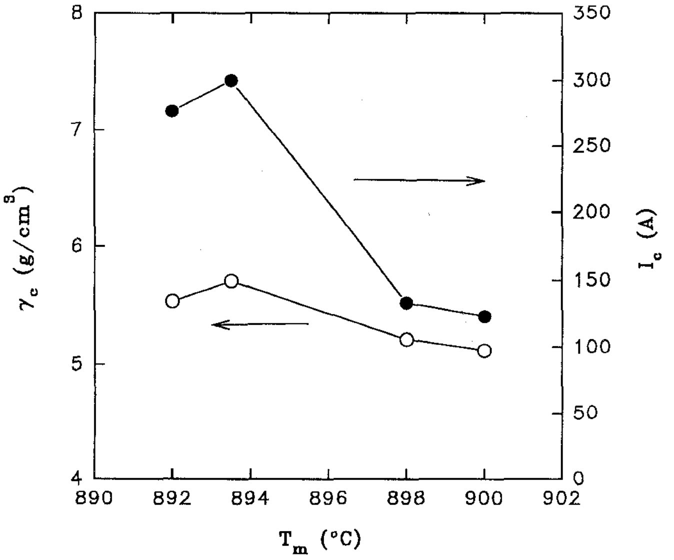

Fig. 4 The density $\gamma_c$ and $I_c$ of the samples as a function of $T_m$. The small volume of our samples (~ $10^{-3}$ $cm^3$) limits the measurement accuracy to 5%.

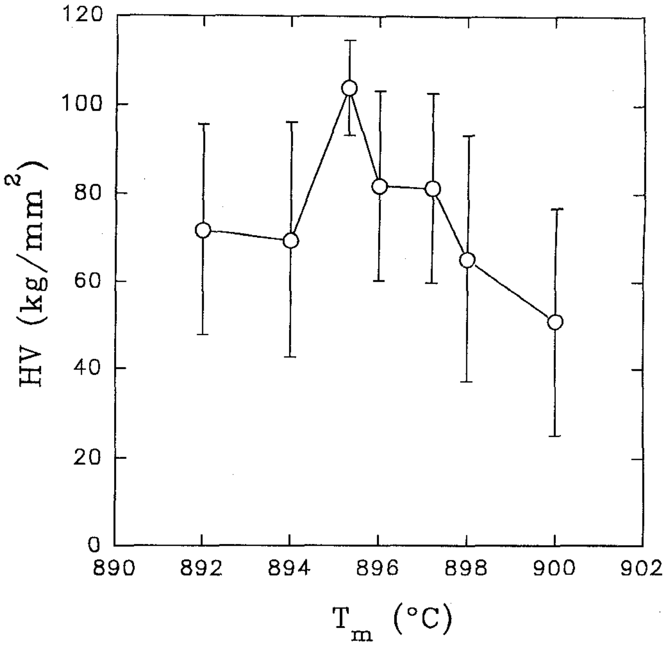

Fig. 5 The mean value of the Vickers microhardness measured at 13 points on longitudinal and transverse cross-sections. The standard deviation defines the error bars.

A detailed study of the correlation between the MO images and microstructure of 2212 tapes shows [8] that the "weak" areas (bright areas in MO images) include macropores and sections with strongly reduced thickness. From the MO images we conclude that the 2212 tapes with the highest $J_c$ have better connections between grains and fewer macropores. Both qualities result in a larger effective cross-sectional area of the tape, thus permitting it to carry more current.

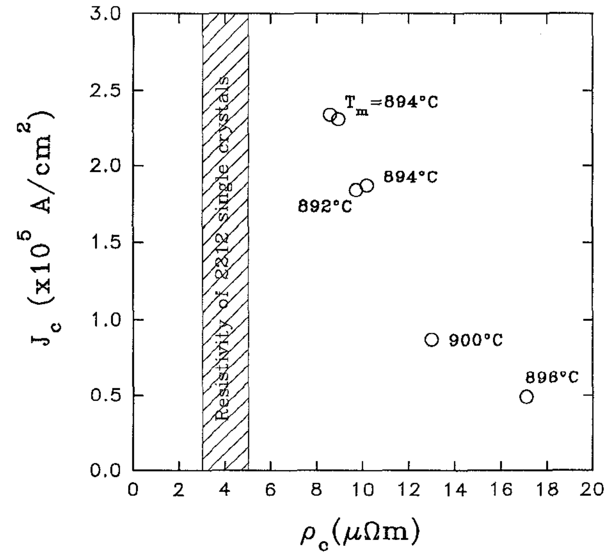

Fig. 6 The room temperature resistivity $\rho_c$ of the BSCCO core plotted against $J_c$ at 4.2K.

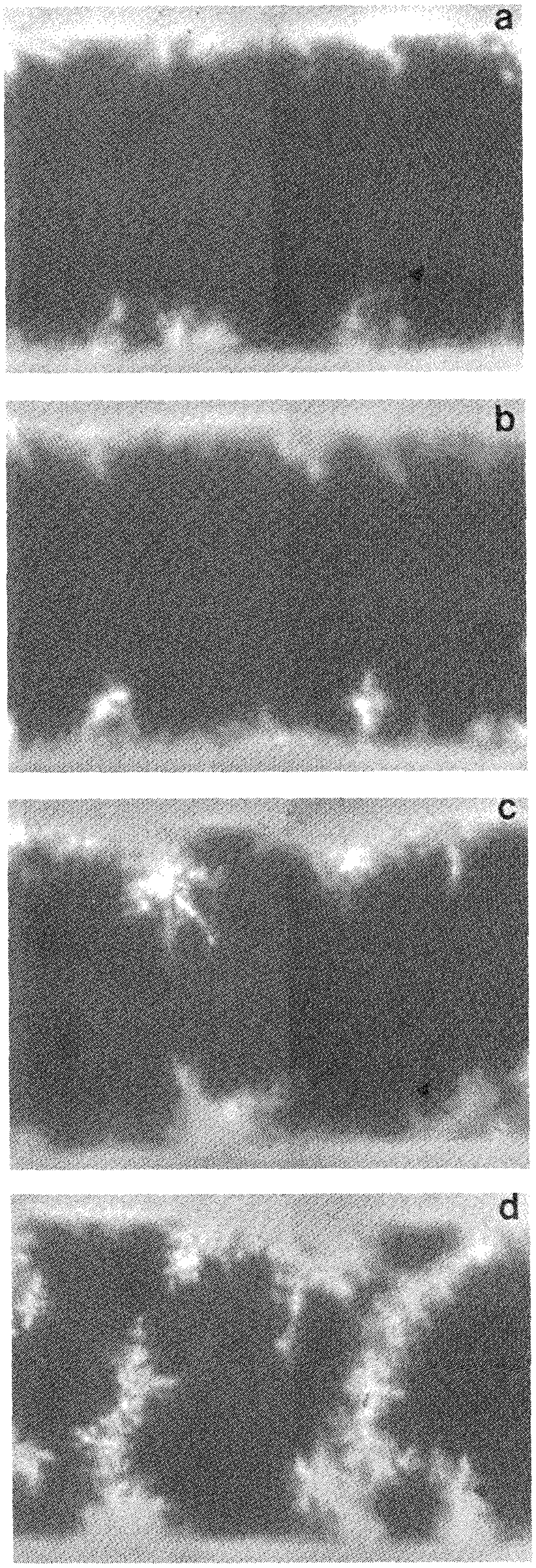

Fig. 7 MO images of the magnetic flux penetration into samples processed at (a) $T_m$=892°C, (b) 894°C, (c) 898°C, and (d) 900°C. The temperature was 15K and the external magnetic field was 20 mT.

## IV. CONCLUSIONS

We studied the influence of $T_m$ on $J_c$ of melt processed Ag-sheathed tapes. For our tapes with nominal composition of $Bi_{2.1}Sr_2CaCu_{1.95}O_x$ , the maximum $J_c$~$2.6x10^5$ A/cm$^2$ ( 4.2 K, B=0, 1μV/cm ) was obtained in samples processed at $T_m$ = 894 to 895°C. A deviation of ±2°C from this optimum $T_m$ reduced $J_c$ by ~25%. No significant changes in the superconducting phase content and grain size for the samples prepared with $T_m$ between 892 and 896°C were found. Enhanced formation of macropores was observed at higher $T_m$. Clearly detectable changes in the density and microhardness of tapes processed at various $T_m$ were observed. Both density and microhardness were the highest for the samples prepared at the optimal temperature $T_m$. In addition, $J_c$ at 4.2 K scaled with the room temperature resistivity. Magnetooptical images of the flux penetration into samples processed with different $T_m$ show that the flux penetration is relatively inhomogeneous. The sample processed at the optimum $T_m$ has better homogeneity and less flux penetration than samples processed at other $T_m$. These results all suggest that $J_c$ is controlled by the effective cross-section of the superconducting core.

## REFERENCES

[1] J. Kase, K. Togano, H. Kumakura, D.R. Dietderich, N. Irisawa, T. Morimoto and H. Maeda, "Partial melt growth process of $Bi_2Sr_2Ca_1Cu_2O_x$ textured tapes on silver," *Jpn. J. Appl. Phys.*, vol. 29, L1096-L1099, 1990.

[2] K. Shibutani, T. Hase, T. Egi, S. Hayashi, R. Ogawa, Y. Kawate, "Fabrication of Ag-sheathed Bi(2:2:1:2) superconducting magnet by means of partial melt and slow cooling process," *Appl. Supercond.* vol. 2, 237-250, 1994.

[3] T. Haugan, S. Patel, M. Pitsakis, F. Wong, S.J. Chen and D.T. Shaw, "Recent status of high temperature superconducting $Bi_2Sr_2CaCu_2O_{8+x}$ wire development at NYSIS: 1-90 meter length $J_c$s and 3 meter diameter ring furnace design" *J. Electronic Materials*, vol. 24, 1811, 1995.

[4] T. Kanai and N. Inoue "Development of gas pressure melting (GPM) method for Ag-sheathed Bi-2212 wires," *J. Mat. Sci.*, vol. 30 , 3200-3206, 1995.

[5] J.A. Parrell, S.E. Dorris, D.C. Larbalestier, "On the role of Vickers and Knoop microhardness as a guide to developing high critical current density Ag-clad BSCCO-2223 tapes," *Physica C*, vol. 231, 137-146 994

[6] E.E. Hellstrom and W. Zhang, "Formation and prevention of bubbles when melt processing Ag-sheathed $Bi_2Sr_2CaCu_2O_x$ (2212) conductors," *Supercond. Sci. Technol.*, vol. 8, 430-438, 1995.

[7] J.A. Parrell, A.A. Polyanskii, A.E. Pashitski and D.C. Larbalestier, "Direct evidence for residual preferentially-oriented cracks in rolled and pressed Ag-clad BSCCO-2223 tapes and their effect on the critical current density," *Supercond. Sci. Technol.*, vol. 9, 393-398, 1996.

[8] M. Polak, W. Zhang. A. Polyanskii, A. Pashitski, E.E. Hellstrom, and D.C. Larbalestier, "Identification of the role of porosity in causing strong local reduction of critical current in BSCCO-2212/Ag tapes prepared by melt process", to be published in *Supercond. Sci. Technol.*