

# REPORT DOCUMENTATION PAGE

Form Approved  
OMB No. 0704-0188

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1. REPORT DATE (DD-MM-YYYY) 27-03-2002		2. REPORT DATE Final Report		3. DATES COVERED (From - To) 01-07-1999 – 30-06-2000	
4. TITLE AND SUBTITLE  Optimization of $J_c$ in BSCCO Tapes				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER N00014-99-1-0323	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S) Eric Hellstrom				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) University of Wisconsin-Madison 750 University Ave. Madison, WI 53706				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) Deborah Van Vechten Office of Naval Research, ONR 312 Ballston Centre Tower One, 800 North Quincy St., Arlington, VA 22217-5660				10. SPONSOR/MONITOR'S ACRONYM(S) ONR	
				11. SPONSORING/MONITORING AGENCY REPORT NUMBER	
12. DISTRIBUTION AVAILABILITY STATEMENT Approved for public release					
13. SUPPLEMENTARY NOTES None					
14. ABSTRACT The study was to determine if the microstructure of melt processed Ag-sheathed $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$ (2212) tape could be improved by crystallizing 2212 from its primary phase field. Ag-sheathed tape was fabricated with BSCCO powder whose composition was in the Bi-2212 primary field. It was melt processed. It was learned when analyzing the melt processed tape that the powder that was synthesized was contaminated with Teflon. It had decomposed and reacted with the BSCCO forming $\text{SrF}_2$ that shifted the overall BSCCO composition out of the primary phase field. There was not time to synthesize uncontaminated powder in the study. Instead, the effect of the heating rate through the melting point of 2212 was studied. It was found that fully processed tape heated through the melting point at $1^\circ\text{C}/\text{min}$ had the most homogeneous microstructure. Faster rates led to porosity in the core or ballooning of the Ag-sheath, while slower rates led to large, remnant nonsuperconducting phases in the fully processed tape.					
15. SUBJECT TERMS BSCCO composition, Ag sheathed BSCCO tapes, high temperature conductors					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			Eric Hellstrom
U	U	U	UU	4	19b. TELEPHONE NUMBER (Include area code) 608-263-9462

20020405 068

Final report  
Optimization of  $J_c$  in BSCCO Tapes  
Grant # N00014-99-1-0323

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March 27, 2002

## Summary

This research was to investigate increasing  $J_c$  in 2212 conductors by improving the microstructure (i.e., eliminating nonsuperconducting phases) by forming 2212 by melt processing from the 2212 primary phase field. In the primary phase field, the 2212 phase would form directly from the melt that is free of crystalline nonsuperconducting phases. In the actual study, the powder we synthesized was contaminated with F from the Teflon filter we used in the synthesis. We switched to Teflon filters because the silver filter we previously used to make the powder was no longer manufactured. The F present in the melt was tied up as  $SrF_2$ , which changed the overall composition of the liquid. Because this project only had funding for six months of graduate student time, there was not enough time to synthesize new powder. Instead we switched to study the effect of heating rates through the melting point of 2212 on the microstructure of the melt at  $T_{max}$  and fully processed tape. We found three regimes for the heating rate: slow heating ( $0.17^\circ C/min$ ) led to large nonsuperconducting second phases in the melt; fast heating ( $5^\circ C/min$ ) led to bubbles in the melt; intermediate heating ( $1^\circ C/min$ ) gave moderate sized second phases and no bubbles in the melt. Details are provided below.

## Primary phase field studies

We synthesized powder with the composition  $Bi: Sr: Ca: Cu = 1.88: 1.73: 1.23: 2.16$ , which Winnie Wong-Ng and Larry Cook at NIST had calculated was in the 2212 primary phase field. We used aerosol spray pyrolysis, which is our standard method for making BSCCO powder. Prior to this synthesis in this study, we separated the powder from the flowing gas stream using a silver filter, which was a thin sheet of silver that contained micron sized holes. The manufacturer had quit manufacturing the large size silver filters, so we decided to switch to a Teflon filter. After we made the powder, fabricated Ag-sheathed 2212 wires from the powder, and did the heat treatment studies, we found  $SrF_2$  in the BSCCO core. We determined this was due to Teflon that had come off the filter and contaminated the BSCCO powder. The Teflon reacted with the BSCCO powder forming  $SrF_2$  during the powder processing steps in which the nitrates and carbon were removed from the aerosol spray pyrolysis powder.

Our quench studies with the F contaminated powder showed the melt contained crystalline nonsuperconducting phases in the melt. Figure 1 shows the microstructure of a sample quenched from  $881^\circ C$ . One of the phases in the melt is  $SrF_2$ . The presence of

F, which was tied up as unreactive SrF<sub>2</sub>, shifted the overall BSCCO composition, most probably out of the primary phase field. Due to this project only providing funding for six months of graduate student time, there was not time to synthesize F-free powder. Instead we studied heating rates through the melting point of 2212.

### **Studies of the heating rate through the melting point of 2212**

We studied the effect of the heating rate through the melting point of 2212. When 2212 melts, it releases O<sub>2</sub>, which must diffuse through the Ag sheath in Ag-sheathed tapes. If the heating rate is too high, there isn't sufficient time for the O<sub>2</sub> to diffuse through the Ag and the tape can bubble. At very fast rates, these bubbles cause the tape to balloon out, destroying the tape geometry. At slower rates, bubbles can form that are not apparent looking at the outside of the tape, but are seen as internal bubbles (porosity) in the microstructure of the core. We used our normal Ag-sheathed 2212 tape with the overall BSCCO composition of 2.1:2:1:1.95 for the study.

Figure 2 shows the heating rates we used through the melting point of 2212. 2212 begins to melt at 865-870°C. Figure 3 shows the microstructure of fully processed tape processed with different heating rates. We were initially surprised at the microstructure of the slowly heated tape that contained many large remnants of the crystalline nonsuperconducting phases that were present in the melt. However, additional quench studies from T<sub>max</sub> of slowly heated samples showed that these nonsuperconducting phases had already grown large in size during the slow heating. They were large in the fully processed tape because they had not fully reacted during cooling when the 2212 phase formed.

We found that fully-processed tape that had been heated at 1°C/min had the best microstructure. Fortunately, this was the heating rate that we had been using prior to this study, so we have continued to heat through the melting point at 1°C/min.

### **Publications and Presentations:**

No publications came out of this research. The results were presented at Deborah Van Vechten's program review in Cape Cod, MA on March 5-7, 2000.

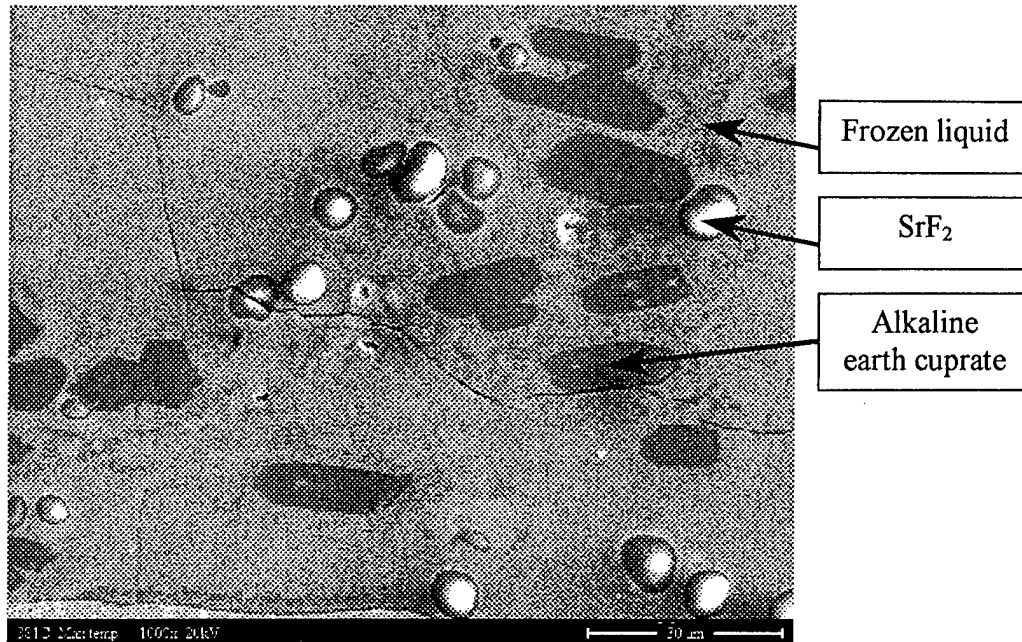


Figure 1. Cross section of the BSCCO core in a Ag-sheathed tape that was quenched from 881°C. The BSCCO powder was initially contaminated with Teflon that decomposed forming SrF<sub>2</sub>. The phases present in the core are frozen liquid, alkaline earth cuprate, and SrF<sub>2</sub>.

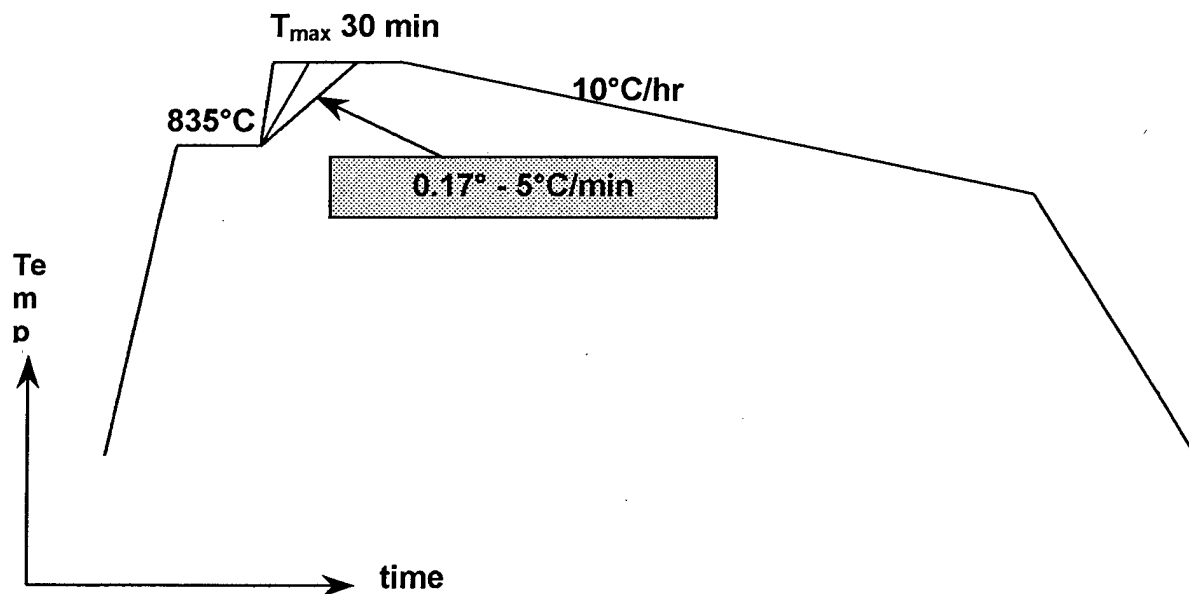


Figure 2. Processing schedule to study the effect of heating through the melting point of 2212 at different rates. 2212 begins to melt between 865-870°C. T<sub>max</sub> = 890°C.

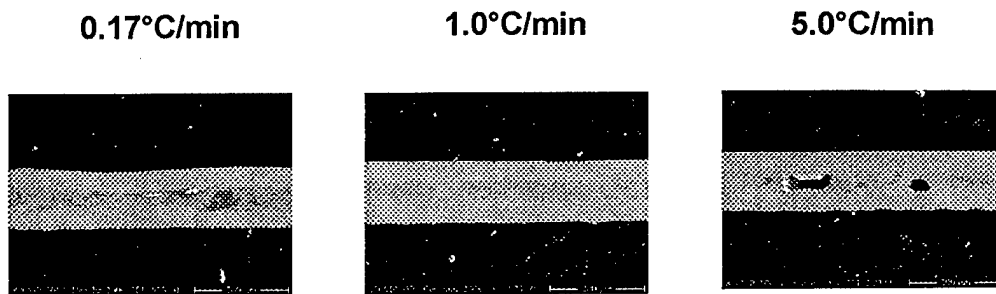


Figure 4: Longitudinal cross sections of fully processed Ag-sheathed 2212 tape with different heating rates. Note the large, remnant, nonsuperconducting phases present in the tape processed with  $0.17^{\circ}\text{C}/\text{min}$  heating. The thickness of the this tape had also decreased. The best microstructure, and presumably highest  $J_c$ , was obtained using  $1^{\circ}\text{C}/\text{min}$ . At  $5^{\circ}\text{C}/\text{min}$ , the fully processed tape contained large pores that had formed when  $\text{O}_2$  evolved during melting. The heating rate was slow enough that the  $\text{O}_2$  that evolved did not cause the Ag sheath to balloon, i.e., the dimensions of the tape had not increased significantly during melting due to  $\text{O}_2$  evolving during melting.