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ANNUAL REPORT

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DEVELOPMENT OF ELEMENTS OF A HIGH T<sub>c</sub>  
SUPERCONDUCTING CABLE

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## **PROGRAM SUMMARY**

The program is aimed at the development of long lengths of silver-clad BSCCO. The material of choice is BSCCO-2223 with 20K the operating temperature goal. Such a tape conductor ultimately could be used in a coil for a magnet, motor or generator.

The program is designed to tackle several key problems with parallel tasks. A large variety of BSCCO powders are under investigation. Silver tubes are packed with superconductor powder and deformed to form tape conductors. An alternate method of making silver-encapsulated superconducting tape is being pursued. Variations in the powders and processing parameters are used to optimize the tape  $J_c$ .

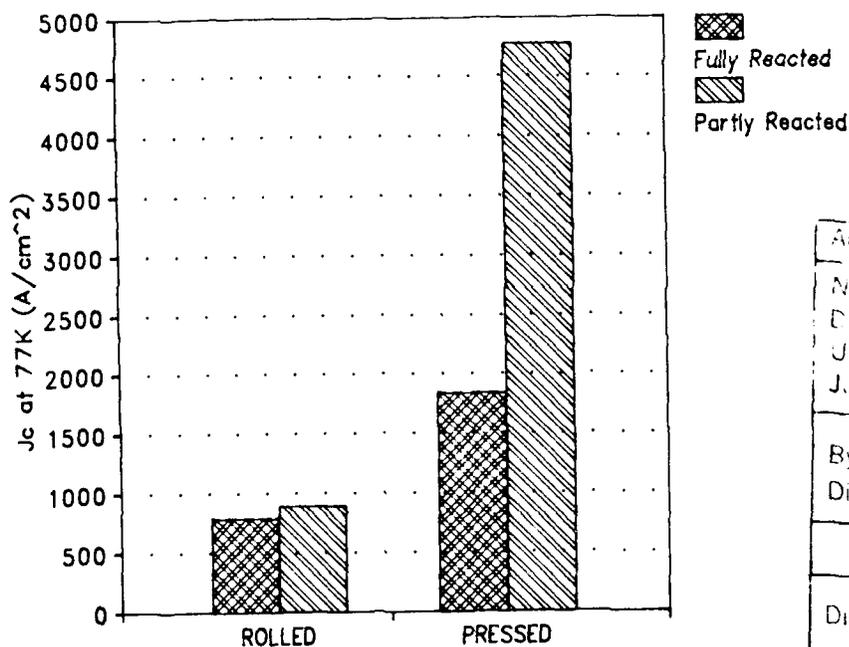
Excellent progress has been made on several fronts as we clarify the conditions required to obtain high  $J_c$  tapes:

- 1) It has been convincingly demonstrated that the highest  $J_c$  tapes are prepared from powder which is not fully reacted to BSCCO-2223. The starting powder should be predominantly the 2212 phase and of a composition which readily reacts to 2223.**
- 2) The superiority of pressing over rolling for the final deformation of the tapes has been proven. The concurrent elongation which occurs during rolling is thought to be detrimental.**
- 3)  $J_c$ 's approaching  $10,000\text{A}/\text{cm}^2$  at 77K and zero applied field have been obtained. The values at 20K and 2T are about 5 times higher.**
- 4) 14 meter long tapes have been made by the PIT process. Longer tapes are possible if larger diameter and/or longer silver tubes are used.**
- 5) A low  $J_c$  degradation of bent tapes allows coiled lengths to be processed. We are now doing final heat treatments of coils on 15cm diameter alumina supports and have demonstrated a technique for pressing long tapes.**
- 6) The alternate silver clad tape process has been simplified with the demonstration that tapes can be made from powder pressed into a silver foil channel. The silver foil package is welded shut with a diffusion bond during tape heat treating.**

## PROGRESS ON TASK 1 -- POWDER PRODUCTION

The goal of this task is the production of reproducible, large powder batches of BSCCO to be used in the tape development effort.

Fully reacted 2223 powders do not result in the best tapes. The highest  $J_c$  values are obtained when the reaction to form 2223 occurs during the final heat-treatment processing of the composite tapes. The initial powder should contain the 2212 phase but with the overall composition designed to readily form the 2223 phase. Figure 1 shows the dramatic effect on critical current density when a partly reacted powder is used. Note also that the effect is much greater for a pressed tape compared to a rolled tape. More details are discussed under task 5.



**Figure 1** Critical current density of BSCCO-Ag tapes at zero applied field. Demonstrates the effect of initial powder 2223 content and final deformation method on  $J_c$ . Tapes were swaged, drawn, and rolled to 0.25mm thickness and heat treated 48hr at 830°C in air. They were then either rolled to 0.125mm or pressed followed by a second identical heat treatment

The ideal powder would be mostly Bi-2212 but would react quickly and completely to Bi-2223. We are pursuing two different approaches. In one the initial powder is a composition known to react readily to 2223 but which is

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only partly reacted before tape fabrication. In the second method, the initial powder is a mixture of fully reacted 2212 and other phases. In both cases we are considering cation ratios differing from the nominal 2223 values. All powders contain some lead substituted for the bismuth to stabilize the 2223 phase. The addition of excess calcium and copper greatly increases the reaction rate.

We are continuing to explore variations on initial powder compositions. Differences in critical current for different powder compositions will be discussed more fully under Task 5. The operating theory is the need for a large volume fraction of 2212 which is mechanically aligned during processing. The overall chemical composition and phase assemblage must react readily to convert the 2212 to 2223.

### **PROGRESS ON TASK 2 -- TAPE FABRICATION USING POWDER-IN-TUBE**

Deformation of BSCCO-filled silver tubes is done by sequential swaging, drawing, and rolling. The tapes are then subjected to final deformation and heat-treatment cycles under task 5 to optimize properties, especially  $J_c$ . The aim of this task is to provide tapes for the  $J_c$  optimization studies.

A process for fabricating tapes as thin as 0.1mm is in-hand. Typically 0.11m long powder columns packed into 6.35mm diameter silver tubes result in 2.7m long tapes 0.25mm thick and about 3mm wide. Further rolling can reduce the tape thickness to 0.1mm if desired. We have made 14 meter long 0.25 mm thick tapes using commercially available 0.5m silver tubes. Longer lengths can be made depending on the availability of longer and/or larger OD starting tubes.

The initial powder to silver ratio determines the ratio in the final tape. We granulate our initial powders to increase the powder filling factor. Using thin-walled silver tubes we have obtained up to 40 volume % superconductor in the tapes which can be compared with typical reported values of about 25%. Swaging is done to densify the powder column, thicken the silver wall and work harden the silver. Most of the diameter reduction and fiber lengthening is done by drawing. A more uniform superconductor thickness across the final tape is obtained if the final drawing operation converts from round to rectangular cross section using a turks head roll set. The diameter at the transition from drawing to rolling controls the final tape width. We typically draw to 1.3 - 1.5mm which gives 2.3 - 2.8 mm wide tapes at 0.25 mm thickness. During the rolling operation the Bi-2212 in the superconductor core is aligned with the basal planes perpendicular to the thickness of the tape.

Since the ultimate test of process variables is the tape  $J_c$ , a large number of tapes have been made. We continue to explore the effect of deformation processing variables on the properties of final tape.

### **PROGRESS ON TASK 3 -- ALTERNATE TAPE FABRICATION PROCESS**

An alternate process for the fabrication of high-Tc conductor Silver-Clad Tape (SCT) is under study. The first step was to have been extrusion of a thin tape containing BSCCO and organic binder. The extruded tape would be partially wrapped in silver foil and heat-treated in an oxidizing atmosphere to remove the organics from the composite tape.

An improvement in the process is feasible. We have demonstrated the ability to fill a silver u-shaped channel with Bi-2223 precursor powder, compress the powder, fold the edges, and diffusion bond the silver-clad package. Such a package has been made and processed by rolling to a thickness of 0.25mm. The superconductor to silver ratio of these composite SCT tapes are in the same range as for PIT processed tapes. This process modification has the advantage over the original concept of not requiring a binder removal step. The length of time required for binder removal from an extruded preform would have made a rapid continuous tape fabrication process difficult.

### **PROGRESS ON TASK 4 -- SILVER FOIL SEALING**

The second step in the GE SCT process is the sealing of the edge of the silver foil wrapped around the BSCCO tape.

The silver foil sealing was to be done using pressure welding. An improvement in the process has been demonstrated. If a lap joint of the silver foil is simply pressed together, the silver cladding diffusion bonds during the superconductor heat treatment process. The bond survives subsequent deformation (rolling or pressing) of the tape.

The SCT process now under consideration involves wrapping silver foil around a packed superconductor powder core and heating to seal the silver edges together. The rectangular cross section silver-clad composite could then be processed in a similar manner to as turks head rolled PIT tape preform.

### **PROGRESS ON TASK 5 -- SUPERCONDUCTOR OPTIMIZATION**

This key task studies the optimization of the  $J_c$  of silver-clad BSCCO tapes.

Emphasis in this task continues on optimization of heat treatment and deformation conditions to maximize  $J_c$ . Many iterations are required to determine the best set of processing parameters. This involves choice of the correct powder as well as the optimum deformation and heat treatment cycles.

Transport  $J_c$  measured at 77K with no applied field is used as a rapid measure of sample quality. The critical current at 20K in an applied field is approximately given by  $I_c(20K, 2T) = 5 I_c(77K, 0T)$ . More detailed measurements as a function of temperature and field can be done on

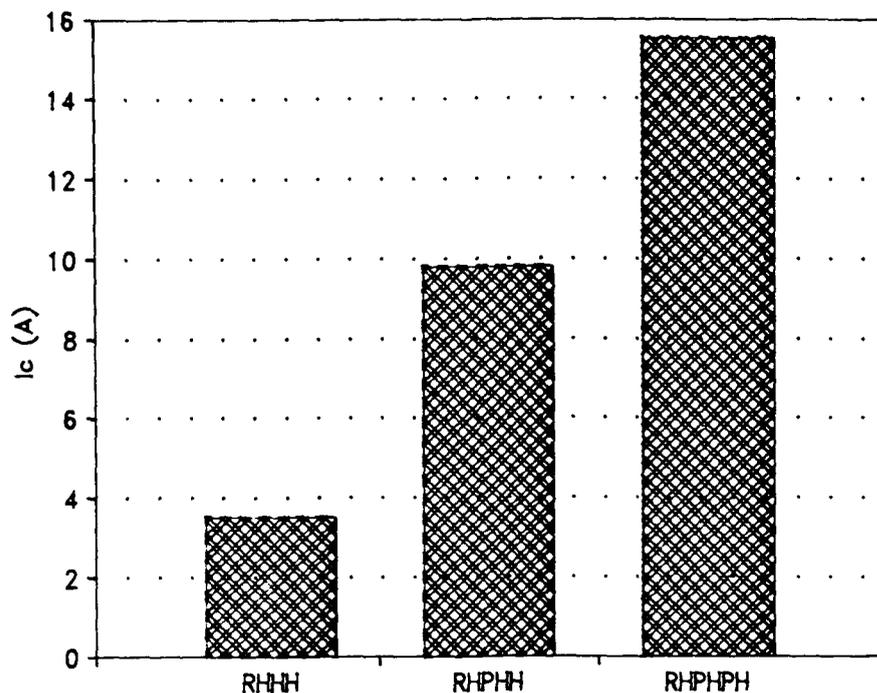
selected samples. Short samples and small coils can be tested at 4.2K with currents up to 1000A and fields up to 10T. A variable temperature apparatus is capable of measuring samples at up to 240A at fields up to 9T.

The final processing of PIT tapes involves a series of heat treatment and deformation cycles. The optimum heat treatment temperatures (in air) for our compositions are in the range 825 - 840°C. Higher temperatures drastically degrade the samples by a partial melt involving the silver sheath. Lower temperatures do not develop the necessary well-sintered 2223 phase.

We have conclusively shown that fully reacted 2223 powder may not be desired. Results of a parallel GE CRD contract with ORNL showed that high  $J_c$  values in PIT tapes could be obtained starting with 2212 powders and reacting to 2223 after fabrication of the tape configuration. We have expanded on this technique and have shown that good  $J_c$  values are obtained on tapes made from partially reacted powders. The initial powder can either be a partially reacted mix which has not yet been totally reacted to Bi-2223 or it can be a mixture of pre-reacted Bi-2212 and other phases. It is important that the precursor powder consist of principally Bi-2212. This phase is aligned during the mechanical deformation processing of the silver plus superconductor composite. The reason for the superiority of partially reacted powder versus fully reacted Bi-2223 is now under study. It seems likely that more grain growth and better intergrain connectivity is obtained by "reaction sintering" since sintering and grain growth of Bi-2223 is very slow.

The choice of deformation modes used during the final processing of tapes has been shown to be crucial. We have shown that pressing is much superior to rolling to obtain high  $J_c$  values.

The rolling or pressing operations are needed to densify the superconducting core. Not only does no densification occur during the reaction sintering operation, the superconductor expands (de-densifies). Figure 2 shows the improvement in critical current obtained by pressing the tape twice as compared to a single pressing and no pressing at all.



**Figure 2** Critical current of BSCCO-Ag tapes at 77K and zero applied field for different final processing conditions. R = Rolled to 0.25mm. H = Heated 48hr at 830°C. P = Pressed at 965MPa.

The difference in the two deformation modes explains the different  $J_c$  values obtained. During rolling of the silver-clad composite the superconductor sees a longitudinal tensile stress which is evidenced by the tape elongation. The in-plane deformation during pressing is confined to widening of the tape. Frictional forces at the punch faces prevent elongation. Any cracks or grain separations developed during rolling will be transverse to the tape while possible cracks from pressing will be along the tape and parallel to the current flow. We have built a special pressing jig to allow semicontinuous pressing of long tapes.

Figure 3 shows the effect of different amounts of excess calcium and copper oxides on the critical current of tapes after a total heat treatment time of 96 hours. The two tapes with the highest excess oxides reached critical current levels of about 15 Amperes after a second pressing and an additional 48 hours of heat treatment. It seems that the different compositions can influence the rate of the reaction, and therefore the heat treatment time needed to get high  $I_c$  values, but the final  $I_c$  attained is relatively independent of the composition.

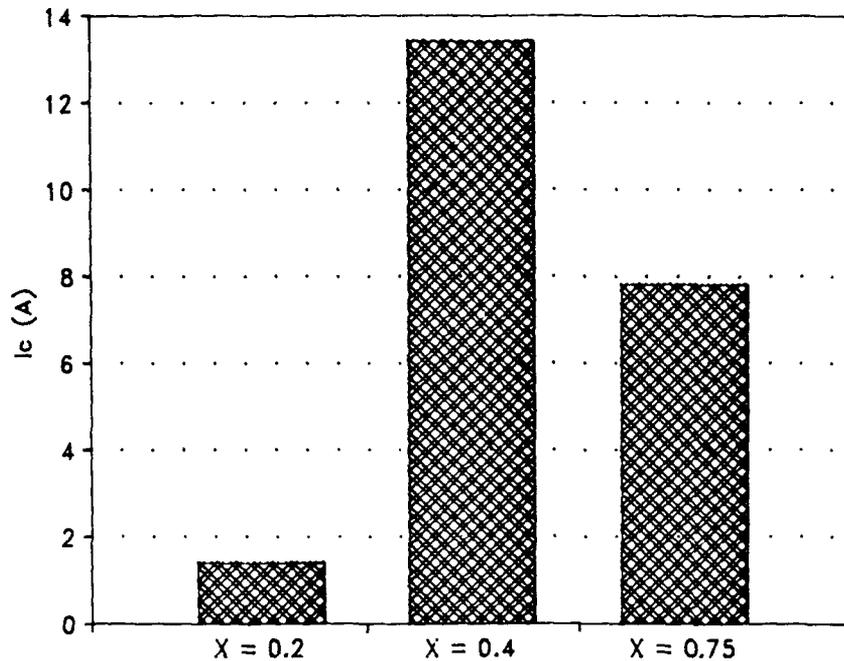


Figure 3 Critical current of BSCCO-Ag tapes at 77K and zero applied field for three different amounts of excess Ca and Cu oxides in the initial, partially-reacted powders. Powder compositions =  $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_{2+X}\text{Cu}_{3+X}\text{O}_Y$

### PROGRESS ON TASK 6 -- LONG LENGTH AND SINGLE COIL PROPERTIES

The properties of long lengths will be studied using coiled tapes. Uniformity along the tape is particularly important.

A necessary condition for the processing of long lengths is the ability to coil the tapes so that the heat-treatments can be done in a batch mode. Tapes with thicknesses as high as 0.25mm total thickness must be bent around radii small enough to allow coils to be placed in small furnaces. In addition the final tapes must have reasonable bend tolerances to allow small coils to be made. We found that 0.25mm thick tapes could be bent around a 3" radius with little degradation in I<sub>c</sub>.

We have done final processing (sequential heat treating and pressing) on 0.22mm thick tapes as long as 1.1m. We are now perfecting final processing techniques for long tapes. Lengths about 1.1m long have been coiled on 15cm diameter alumina cylinders for heat treatments. A pressing technique where 15.2cm segments of the plate are sequentially pressed with about 1.3cm overlaps between pressings. This semicontinuous pressing

operation is amenable to automation. No degradation of  $I_c$  is seen at the overlap joints.

### **TALKS AND PAPERS**

KW Lay, "Critical Currents in Aligned YBCO and BSCCO Superconductors", Invited talk given at New York State Institute on Superconductivity Conference, Sept. 20, 1990, Buffalo, NY, Published in Superconductivity and its Applications, Y-H Kao Ed., AIP, NY, 1991, pp. 111-129

KW Lay & JE Tkaczyk, "Material and Process Choices for Long High  $T_c$  Tapes and Wires", Talk given at DARPA Meeting in Danvers, MA, Oct., 1991

JE Tkaczyk,, RH Arendt, JA DeLuca, MF Garbaskas, PL Karas, KW Lay, A Mogro-Campero, LG Turner, "Improved Magnetic Field Dependence of  $J_c$  in c-axis Aligned Polycrystalline High Temperature Superconductors", Talk given at Materials Research Society 1990 Fall Meeting, Boston, MA, Nov 29, 1990

KW Lay, "C-Axis Texturing in High  $T_c$  Superconductors", Talk given at DARPA Workshop on HTS Bulk Technology, Santa Fe, NM, 1/31/91

JE Tkaczyk,, RH Arendt, PJ Bednarczyk, MF Garbaskas, KW Lay, "Transport Critical Current Measurements of Bi-Sr-Ca-Cu-O High Temperature Superconductors", Talk given at American Physical Society Spring Meeting, Cincinnati, OH, March, 1991

### **GOALS FOR NEXT PERIOD**

Determine the optimum initial powder phase assemblage to minimize reaction sintering time.

Continue evaluation of alternate fabrication method using powder packed in a silver channel.

Quantify the factors influencing  $J_c$  of tapes.

Fabricate longer tapes and determine their critical currents.

## FINANCIAL STATUS

All values are cost plus fixed fee total costs.

TOTAL FUNDING REQUIRED FOR EFFORT	\$2,424,530
01Sept88 through 31Dec91 (40 months)	
CURRENT AUTHORIZATION	1,668,000
01Sept88 through 31Jan91 (29 months)	
FUNDING EXPENDED TO-DATE	1,885,840
01Sept88 through 30Jun91 (34 months)	
ESTIMATED THROUGH END OF GOV'T FY91.....	2,190,000
01Sept88 through 30Sept91 (37 months)	