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Progress Report
on
**GROWTH OF HIGH T_c SUPERCONDUCTING FIBERS USING
A MINIATURIZED LASER-HEATED FLOAT ZONE PROCESS**
for the period
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13. ABSTRACT (Maximum 200 words) This report covers the research accomplished on the program entitled "Growth of High T_c Superconducting Fibers using a Miniaturized Laser-Heated Float Zone Process" during the 12.5 month period from Oct. 15, 1989 to Nov. 5, 1990. Research was done in four areas: phase relationships, preparation of starting materials, growth studies and the advanced fiber growth apparatus. The phase relationship studies built on the work published by Ono. Comparison studies with the well known compound $\text{Ca}_3\text{Al}_2\text{O}_6$ confirmed that the $\text{Bi}_{2+x}(\text{Sr,Ca})_{3-x}\text{Cu}_2\text{O}_y$ is incongruently melting and that cuprous oxide, calcium oxide and (strontium,calcium) cuprate are the higher melting compounds which coexist with the melt and the superconducting phase. The preparation of the starting materials is crucial to the stable growth of the fibers. Non-uniform distribution of second phase particles, gaseous inclusions or porosity can lead to instabilities. A process was developed to ensure uniform starting materials. This process involves grinding the individual starting materials to a uniform size ($44\mu\text{m}$). The resulting powders are mixed and calcined three times with regrinding between each calcining step. The calcined powder is then cold pressed and sintered, reground, re-pressed and sintered. This final material is then cut into bars for feed material for fiber growth. Growth rate studies showed a relationship between the growth rate and the regions of stability for single and multiphase fibers. This was traced to changes in the Bi and Cu levels in the melt related to changes in the growth rate. It was also shown that fluctuation in laser power lead to CaO inclusions in the fibers. The necessary components for the Advanced Fiber Growth Apparatus have been determined. Some of the components have been ordered and others are being designed.				
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GROWTH OF HIGH T_c SUPERCONDUCTING FIBERS USING A MINIATURIZED LASER-HEATED FLOAT ZONE PROCESS

I. INTRODUCTION

The primary objective of this three-year program was to establish the viability of the laser-heated pedestal growth (LHPG) method, a miniaturized float zone process, for the economical preparation of superconducting wires from the new class of high T_c superconducting copper oxide ceramics. These new materials have, since their recent discovery, excited the imagination and interest of scientists world-wide, and important applications have been envisaged. It is not surprising, however, that many important questions have arisen concerning their suitability for certain applications. These include not only whether they have the properties required, but also whether economical, effective processing techniques can be developed. Extensive efforts to produce both thin films and wires are underway in many laboratories, the former for electronic devices and the latter for motor, magnet and power transmission applications.

This program has concentrated on one of the new types of high T_c copper oxide based superconductors $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$. It exhibits high transition temperatures and high critical currents and also has better thermodynamic properties (low volatility in particular) compared with other superconducting materials. This superconductor, however, may be more susceptible to flux creep and poor conductivity in high magnetic fields which could limit its suitability for dc operations. At the present time we do not know enough about the mechanisms involved in the superconducting behavior of these oxide materials, or the relationship between properties, processing conditions and structure to eliminate any of the above from consideration. We are keeping abreast of developments and are modifying the program accordingly as new information becomes available.

Most of the current work to develop wires of the new high T_c superconductors has concentrated on polycrystalline ceramic samples having grains with random crystallographic orientations and intergranular barriers between the grains which have limited critical current densities to much less than the highest values which can be obtained with single crystal samples. While numerous, relatively inexpensive ceramic processing techniques are currently under study for making long lengths of polycrystalline superconducting wires, the ability to overcome the above problem has still not been achieved. Grains which can be continuously aligned so that the appropriate current carrying planes are directed along the wire axis (c-axis normal), or single crystal wires will eliminate these "weak links" (grain boundaries) mentioned above.

Preliminary experiments (1,2) carried out during the last few years on the growth of the Bi containing superconductors using the LHPG method have yielded very encouraging results, including fibers with transition temperature onsets of 105°K, zero resistance at 85°K, minimum critical current densities of 40,000 amps/cm² at 68°K and flexibility in fibers 100 μm in diameter or smaller. These fibers, having the nominal composition $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ (2212), have an oriented grain structure with the current carrying planes parallel to the fiber axis and superior properties compared to wires produced by ceramic processing techniques. At slower growth rates single crystal fibers can be produced which do not exhibit intergranular barriers that can impede current flow.

For superconducting motor, magnet or transmission line applications, long lengths (kms) of small diameter fibers capable of carrying large critical currents at temperatures above 77° K are needed. While the fibers grown by the laser-heated float-zone growth method have been shown to possess excellent superconducting properties, an important concern is whether these fibers can be made in long lengths and small diameters. Fibers have been grown as small as 25 μm in diameter (1cm in length) and in lengths up to 14cm (100 μm in dia.). The prospects look very good for dramatically increasing the lengths of

even the smallest diameter fibers with appropriate modifications to the LHPG equipment and growth technique.

Critical issues which are being considered during the course of this program include: 1) the most appropriate compositions which can yield long lengths of uniform structure and composition with high superconducting transition temperature, T_c , and critical current densities, J_c , 2) the maximum allowable growth velocity which can be used before degradation of the fiber properties, and 3) the maximum length of fiber which can be produced. To address these issues, this program involves an in-depth study of 1) the thermodynamic and kinetic factors which affect growth rates and the properties of the fibers produced, 2) the development of a dedicated advanced fiber growth system which will permit better control of system parameters, and 3) the development of techniques to enhance fiber throughput via increased growth velocity and the growth of many fibers simultaneously.

II. PROGRESS TO DATE

During the 12 month period covered by this report the emphasis has been on 1) improving the quality of the ceramic starting material, 2) developing techniques for the growth of small diameter fibers, 3) increasing the growth velocity of single phase fibers and 4) set up of the new growth system. In order to solve some of these problems a more thorough knowledge of the phase relationships in the Bi-Ca-Sr-Cu-CuO system has been necessary.

Phase Relationships

The bismuth alkaline earth cuprate 2-layer superconductor is incongruently melting. In previous work, it was found that growth from stoichiometric starting material ($\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_4$) produced fibers which had single phase regions of a different stoichiometry from the starting material. These fibers were subject to sudden growth instabilities which disrupted the continuous formation of superconducting material. It was theorized that stable single-phase fibers could not be grown from these compositions for long periods because they were compositions which were not in thermodynamic equilibrium with the melt. Therefore, a study was undertaken to determine starting compositions from which superconducting, single phase, inclusion-free $\text{Bi}_{2+x}(\text{Sr,Ca})_{3-x}\text{Cu}_2\text{O}_4$ fibers could be grown after the initial transient phase of growth is past.

Published work by Ono [1] has determined that the nominal " $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ " superconducting phase has a range of stability which does not include the exact 2212 stoichiometry. Figure 1 shows the various compositions tested in our study. These are shown to be either within or just outside Ono's solubility limits. The filled circles indicate compositions which could be used to produce stable single phase fiber growth and the open circles indicate compositions which could not.

Information concerning the details of the solidification process was obtained by first quenching melts during steady state growth (using different composition starting material) and then analyzing the fibers and their quenched melts. Optical pyrometry was used to determine the approximate melting temperature of different compositions of the superconductor as fiber growth occurred. Polished lengthwise cross-sections of the fiber, quenched melt and the source rod, were examined by microprobe analysis to evaluate the composition profile of the fiber and the composition of the melt from which it was grown. Examination of the melt-source rod interface in these samples also revealed ways in which the source material could contribute to growth instabilities.

Four gold contacts were applied to the polished cross-sections so that R vs. T curve could be obtained for samples of known chemical composition and homogeneity. Thus we

were able to evaluate the effect of chemical composition and homogeneity on the superconducting properties of the fibers.

Fig. 1 Stability - Composition diagram after Ono (1) showing compositions explored in this study

Analysis of the fibers and their quenched melts showed that these fibers were superconducting, as grown, with $T_c(R=0)$ between 76 and 87°K and that they grew from melts that are Bi_2O_3 -rich and SrO and CaO -poor.

In order to evaluate the correspondence between the solidification conditions observed for the superconducting fibers and the phase equilibria in the Bi-Sr-Ca-Cu-O system, similar fiber growth experiments were performed on $\text{Ca}_3\text{Al}_2\text{O}_6$ a well known material. Both this compound and the superconductor undergo a similar peritectic decomposition on melting. These showed that the melt compositions of off-stoichiometric fibers could be predicted from established phase equilibria and explained some of the fiber morphologies we had been seeing in growth of the superconducting fibers. As a result of these studies, it was verified that the $\text{Bi}_{2+x}(\text{Sr,Ca})_{3-x}\text{Cu}_2\text{O}_y$ phase is incongruently-melting and that Cu_2O , CaO and $(\text{Sr,Ca})\text{CuO}_2$ are higher melting compounds which coexist with the melt and $\text{Bi}_{2+x}(\text{Sr,Ca})_{3-x}\text{Cu}_2\text{O}_y$.

Preparation of Starting Material

The effect of processing parameters on the microstructure of the starting material was actively investigated during this phase of the program. The goal was to determine the proper fabrication temperatures, calcination and sintering times, grain size, pressing-pressures, and starting composition in order to achieve dense, homogeneous starting materials. Having a dense, chemically homogeneous starting material is of critical importance to the success of our program. One of our primary goals is aimed at achieving 30 μm long, flexible fibers, and we reported last year that the growth of such small diameter fibers was postponed until sources material of adequate compositional and dimensional uniformity became available. The growth of these fibers requires the use of small diameter

source rods. Major growth instabilities can occur during such growth due to the presence of non-uniform distribution of second phase particles, gaseous inclusions or porosity (as depicted in Figure 2).

Fig. 2. Examples of perturbations in fiber growth caused by a) solid inclusions b) porosity, and c) swelling of source rod.

Fig. 3. Backscattered electron photograph of a fiber grown from $\text{Bi}_{2.1}\text{Sr}_{1.8}\text{Ca}_{1.1}\text{Cu}_2\text{O}_8$ source rod.

The effect of starting composition was vigorously investigated. As mentioned above Ono [1] reported that single phase 2212 ceramic samples can only be prepared from compositions containing Bismuth in excess of the stoichiometric amount. This extra Bi presumably goes onto the alkaline earth sites replacing strontium or calcium, i.e., $\text{Bi}_2(\text{Bi}, \text{Sr}, \text{Ca})_3\text{Cu}_2\text{O}_8$. The single phase region is also affected by the Ca/Sr ratio. Based on Ono's work, we were able to produce more uniform source material and thereby fibers with more uniform diameter and composition than those grown from the stoichiometric 2212 starting composition.[2] Figure 3 shows a back scattered electron

photograph of a fiber grown from $\text{Bi}_2, \text{Sr}_{1.8} \text{Ca}_{1.1} \text{Cu}_2 \text{O}_8$ source rod at constant laser power.

The processing parameters for the fabrication of starting material were carefully identified and optimized. Appropriate purity, grain size and preconsolidation (cold-pressing) are vital if reproducible results are to be achieved. Contamination and non-uniformity during powder processing will be carried throughout the subsequent processing steps leading to deficient starting material.

Optimum calcination conditions were found which yield better reacted powders. This in turn gave us less gas entrapment in the final starting material. Uniaxially cold-pressing was done at a variety of pressures (5,000-100,000psi range), and Fig. 4 shows the green density as a function of pressing pressure. (Density after sintering follows a similar pattern). Sintering temperatures as well as sintering times were also investigated and optimized.

Fig. 4 Green Density as a function of pressing pressure.

Careful control of particles size in each stage of the fabrication process was achieved by screening the powders down to a 325 mesh (44 μm particle size).

The optimized process can be summarized as follows:

The initial powders were ground in an agate mortar and sieved. Stoichiometric amounts of these oxides were weighed and carefully mixed using the agate mortar. The mixed powders were then subjected to a three-calcination step procedure. i) 760° C for 24 hrs; ii) 800°C for 24 hrs and; iii) 840°C for 48 hrs. After each step, the powder was re-ground and sieved. After that, the samples were uni-axially cold pressed into round pellets. The pressed pellets were sintered for 2 days at 860°C. The sintered samples were then reground, sieved, re-pressed and sintered again under the same conditions. Morphological studies of these starting materials were done using SEM and X-ray diffractometry.

Figure 5 illustrates the growth of fibers from (a) our presently "good" single-phase $\text{Bi}_{2.1} \text{Sr}_{1.8} \text{Ca}_{1.1} \text{Cu}_2 \text{O}_2$ starting material and (b) "bad" (inhomogeneous, porous)

Fig. 5 Growth of fibers from (a) "good" source material, (b) "bad" (inhomogeneous, porous) source material

starting material. As easily can be inferred from these pictures, we have achieved a more stable growth with better diameter control and no gaseous inclusions in the growing fiber.

Growth Studies

The relationship between growth rate R , fiber diameter D and the formation of single crystal, polycrystalline single phase and multi phase fibers have been carefully defined, as shown in Fig. 6.

Fig. 6 Growth rate versus fiber diameter plot showing stability regions for single crystal, single phase and multiphase fibers.

From constitutional supercooling arguments, when the ratio of thermal gradient G to growth rate R is larger than a critical value $(G/R)_c$, flat interfaces (single crystal growth) and single phase growth can be achieved. Since G increases with decreasing fiber diameter (calculated), as seen in Fig. 7, smaller diameter fibers may permit higher growth rates. This explains why the transition from single crystal to polycrystalline single phase growth

Fig. 7 Variation of axial temperature gradient with fiber diameter.

behaves as shown in Figure 6. From calculations, we have found that $(G/R)_c = 1.0 \times 10^{12}$ KS/M², as described in Ref. (3).

Recent experiments have shown that at slow growth rates, the composition of the molten zone contains Bi_{2.47} Sr_{1.34} Ca_{1.02} Cu_{1.93} O₈, (i.e. Bi rich). At higher growth rates, the Bi level in the melt is lowered and the Cu level increased. Similar results were obtained by M.J. Cima et al in MIT. We believe this change in Bi and Cu levels in the melt causes the transition from the single phase to multi phase growth region seen in Fig. 6.

While it is difficult to find single crystal seed material, we have found that Pt wire can be used very effectively. It is better than Au wire and seeds cut from conventional ceramic source material. The initial crystallites nucleate on the tip of the Pt wire, and grow along the fiber axis. After a length 4 times the diameter, grain selection generally leads to the formation of only a few grains dominating the fiber cross-section, as shown in Fig. 8. Such textured structures with strong grain alignment are desirable for current carrying capacity.

Experiments have shown that laser power fluctuations can introduce CaO inclusions into these fibers. Low frequency fluctuations will disturb the grain structure. Using high quality source rods, the longest fiber length that has been grown is 14 cm ($d=90\mu\text{m}$). This fiber can be bent to a radius of ~ 5 cm, as shown in Fig. 9. The smallest diameter we have grown to date is $10\mu\text{m}$ (~ 2 cm long), as shown in Fig. 10. A cross section of a typical 2-5 grain fiber shows a micaceous structure (Figure 11).

Annealing has been found to be a useful technique for raising T_c and J_c (Ref. [2]) in multi phase fibers. Annealing of single phase fibers may make the existing high J_c even higher. Research is underway to explore the relationship between annealing conditions and superconducting properties.

III. ADVANCED FIBER GROWTH APPARATUS

A major objective of this program is to design, build and incorporate into the high T_c fiber research a dedicated fiber growth system capable of allowing routine preparation of 10-50 μm diameter fibers of uniform diameter and in long lengths. Improvements in laser stability and focusing optics, diameter control, fiber and source rod mounting and guiding, mechanical stability, and the incorporation of motor drive systems with greater precision are of greatest concern. Several different approaches are possible in each area and the design phase has involved a thorough evaluation of each alternative to identify those that will best meet the goals of this program. Since there are only a handful of LHPG systems in the entire world, each built by a different individual(s), there really

does not exist a proven design for this type of apparatus. We have therefore undertaken to prioritize our system design requirements, modify or design from scratch components when our own unique needs cannot be met by existing apparatus, and integrate them into a complete system.

Fig. 8 Grain structure resulting from the use of platinum wire.

Fig. 9 Superconducting fiber 90 μ m in diameter, 14 cm long bent over a 5 cm radius.

200 μm

Fig. 10 A 2cm long 10 μ m diameter fiber.

Fig. 11 Multigrain fiber showing mica-like layered structure

The first year of this program began with a study to establish design priorities and survey existing LHPG facilities in the U.S. Following that, a preliminary design was generated and thorough evaluations of the two most critical system components, the cw CO₂ heating laser and the precision translation stages, were carried out. Of all the commercial CO₂ lasers currently in production, only one model from Laser Photonics, Inc. was found that offered the output power level and power stability that we felt was necessary for this project.

This particular 25 watt cw CO₂ grating-stabilized sealed waveguide laser has been under evaluation for long term power stability and grating feasibility by the vender since October, 1989. Early in 1990 the vender was able to supply acceptable output recordings from engineering prototypes and an order was finalized in May, 1990. Production difficulties of an unrelated nature have been encountered by the manufacturer and a delivery date is still pending.

Much of the subsequent optical train design hinges in having the laser in hand and functioning. This portion of the project has, therefore, been seriously impacted by the protracted delays in obtaining the laser.

In other areas, a custom optical table and the custom diamond-turned copper focusing optics have been acquired, and custom machining of the various mechanical assemblies is in progress. Acquisition of the custom vacuum chamber has been postponed until 1991. The rest of the system can be assembled and tested without this component, and it was felt that it would be a logical choice to ensure that unanticipated mechanical problems not arise.

Table I reiterates the planned LHPG system components and Table II summarizes the current status of major component acquisition.

TABLE I
LHPG SYSTEM BREAKDOWN

1. Laminar Flow Clean Area
2. Fiber Preparation/Evaluation
 - a. Microscope
 - b. Gauging and mounting
3. Operating Console
 - a. Microprocessor interfacing
 - b. Power monitoring and control
 - c. Process monitoring and control
 - d. Growth chamber ambient control
4. Fiber Monitoring
 - a. Optical monitoring
 - b. Video monitoring and recording
 - c. Video analysis
5. Vibration Isolation Optical Table
 - a. Optical train
 - b. Growth chamber
 - c. Loading and unloading chamber access
 - d. Microscopic viewing reference
6. Vacuum, Purge Gas, and Laser Cooling Support

TABLE II
MAJOR HIGH T_c LHPG SUBSYSTEMS

Item	Description	State-of-Art	Source	Purchase	Use
CO ₂ Laser	cw, ultrastable in pointing stability, wavelength and power. 25 watts req'd. Dither stabilized.	yes	Laser Photonics	1990 (Pending)	Immediate
Optical Focusing	Diffraction-limited focus in a 20μm band around a 30μm dia fiber. Circular symmetry highly desirable	yes	Pneumo-Precision, Inc. (Custom diamond-turned copper optics)	1990	Immediate
Mechanical Translation	Ultrastable feed and pull stages for moving material into and out of the molten zone.	yes	Micro-Mo Inc. (Miniature dc phase locked gearhead motor with computerized control)	1989	Immediate
Optical Monitoring	Diffraction limited monitoring of 20μm x 30μm molten zone from 200mm distance. Large numerical aperture may be necessary.	yes	Wild-Heerbrugg, Ltd. (Stereo-zoom, long working distance microscope.) or Questar, Inc. (Short working distance telescope)	1990	Late 1990
Optical Table	Vibration isolation and mounting of optical train and growth chamber. 8"-10" through port required.	No	Oriel (Custom 30"x48" table)	1990	Early 1989
Optical Beam Handling	Direct laser beam, attenuate and merge with alignment laser beam	No	Newport	1990 (To be assembled)	Late 1990
Mechanical Positioning	Intermittent X, Y, Z positioning	No	Oriel	1989	Late 1990

MAJOR HIGH Tc LHPG SUBSYSTEMS, continued

Item	Description	State-of Art	Source	Purchase	Use
Environ- mental and Ambient	Vacuum-capable growth chamber with controlled atmosphere purge and slight, over pressure capable.	No	Huntington Vacuum	1991	Mid 1991

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