Texturing of High T_c Superconducting Polycrystalline Fibers/Wires by Laser-Driven Directional Solidification in a Thermal Gradient

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ABSTRACT

This paper summarizes the technique of laser-driven directional solidification in a controlled thermal gradient of yttria stabilized zirconia core coated Y-Ba-Cu-O materials to produce textured high T_c superconducting polycrystalline fibers/wires with improved critical current densities in the extended range of magnetic fields at temperatures greater than 77K. The approach involves laser heating to minimize phase segregation by heating very rapidly through the two-phase incongruent melt region to the single phase melt region and directionally solidifying in a controlled thermal gradient to achieve highly textured grains in the fiber axis direction. The technique offers a higher grain growth rate and a lower thermal budget compared with a conventional thermal gradient and is amenable as a continuous process for improving the J_c of high T_c superconducting polycrystalline fibers/wires. The technique has the advantage of suppressing weak-link behavior by orientation of crystals, formation of dense structures with enhanced connectivity, formation of fewer and cleaner grain boundaries, and minimization of phase segregation in the incongruent melt region.

I. INTRODUCTION

Progress in the application of high critical temperature (T_c) ceramic superconducting polycrystalline fibers/wires (with zero resistance above liquid nitrogen temperature at 77K) for very high speed system interconnects, integrated optical components and passive microwave devices for microelectronics and instrumentation applications is currently limited by the lack of formability, strength, toughness and low transport current properties. The low critical current densities (J_c) are associated with extremely small coherence length, causing grain boundaries to act as Josephson junctions such that tunneling currents are highly field dependent; a high degree of superconductivity anisotropy, severely suppressing tunneling currents at high-angle grain boundaries; grain boundary segregated phases; and anisotropic thermal contraction and microcracking at the grain boundaries. The causes of the weak link behavior can be greatly diminished by development of a process method which will yield formation of dense structures with enhanced connectivity, crystallographic texturing of the grains, formation of cleaner and fewer grain boundaries parallel to the conduction direction, and minimization of phase segregation in the incongruent melt zone. One method of forming textured microstructures is directional solidification from the melt in a thermal gradient. However, in the Y-Ba-Cu-O system, the $Y_1Ba_2Cu_3O_7$ or 123 phase could not be obtained congruently from the melt of the 123 composition because of its incongruent crystallization under the equilibrium phase transformation. The 123 phase is formed by the peritectic reaction through the Y_2BaCuO_5 (211) phase and the liquid phase. If the crystal-growth under the metastable state is realized, the peritectic reaction can be suppressed by rapid solidification with very high growth rate due to high interface undercooling and the 123 phase could be crystallized congruently resulting in highly oriented fibers with improved grain boundaries and high J_c. In the 123 phase the critical current density is one order of magnitude higher along the a-b plane than along the c-axis direction.

Research efforts have been directed toward increasing the J_c of high T_c superconducting materials by texturing the grains using different techniques such as melt textured growth, liquid phase processing and directional solidification, quench and melt growth processes, and zone melting and laser float zone melting. All these techniques have resulted in highly textured microstructures (1). The significance of the technique of laser-driven directional solidification in a controlled thermal gradient lies in its adaptability as a continuous process for improving the critical current densities of high T_c superconducting polycrystalline fibers/wires. The technical approach involves laser heating to minimize phase segregation by heating very rapidly through the two-phase incongruent melt region (1010-1258°C) to the single phase melt region (> 1258°C) and directionally solidifying in a controlled thermal gradient to achieve highly textured grains in the fiber axis direction.

II. EXPERIMENTAL TECHNIQUES

Texturing of extruded Y-Ba-Cu-O fiber, Duranickel 301 (95%Ni • 4.4%Al • 0.6%Ti) core coated Dy-Ba-Cu-O and yttria stabilized zirconia (YSZ) core coated Y-Ba-Cu-O high T_c superconducting polycrystalline fibers/wires was investigated. Five cm long fiber segments were mounted on an alumina tube with the help of alumina paste to hold the fiber and translated in a laser-driven directional solidification thermal gradient (LDT) furnace as shown in Figure 1. The computer controlled LDT furnace is comprised of a multiple heater array, a fiber translation mechanism capable of translating at rates of 0.5 to 200cm/hr, a fiber feed and fiber take-up system for feeding the fiber preforms and removing the processed fibers, and a laser beam delivery system consisting of laser optics to homogeneously melt the fiber from four directions to the single phase melt zone. The LDT furnace consists of two split tube furnaces having 6.4cm internal diameter and eight independently computer controlled resistively heated zones. The upper split tube furnace consists of two heating zones, a 6.4cm long binder burnout zone and a 10.2cm long preheat zone. The lower split furnace consists of six heating zones, four 6.4cm long thermal gradient zones and two 10.2cm long annealing zones. Each zone is installed with a temperature sensor to provide flexibility in varying the temperature profile in the thermal gradient. The furnaces are controlled by a closed loop computer based system. A 386-25MHz computer contains two data acquisition and control boards to provide sixteen channels of differential analog input and cold junction compensation for thermocouple signals. An Inconel sheathed type-K thermocouple is installed in the center of each furnace zone and connected to one channel of the expansion system. The DT2815 data translation board provides eight 4-20mA output channels to control each thermal gradient zone using an Omega PCM1 4-20mA driver. Labtech Notebook software controls the furnace zones using a PID control algorithm. Each thermocouple is sampled at a rate of 10Hz. Labtech Notebook also samples a ninth thermocouple at a slower rate and writes the temperature to a data file. By translating this thermocouple through the furnaces, an exact profile of the gradient can be recorded.

The thermal gradient furnace assembly consists of a fiber translation system which includes a friction drive system to feed the fiber into the gradient and to remove the processed fiber (2-3). The LDT furnace is installed with a laser beam delivery system consisting of laser optics to homogeneously melt the fiber from all directions to the single phase melt zone. This is accomplished using an 18 Watt CW Nd:YAG laser beam (λ = 1.06μm) which is evenly divided into 4 x 4.5 Watts, using beam splitters, mirrors and lenses arrangement as shown in Figure 2. The optics are mounted using an adjustable height post system. Each post is fastened to a micro optical rail to maintain alignment between the components. The rails are mounted on an aluminum plate installed surrounding the furnace. A He-Ne laser and four pinhole apertures were used to align each rail so as to focus the 4 x 4.5 Watt laser beam at the center of the laser zone, between the two split furnaces as shown in Figure 1, where the fiber is coaxially translating. All the components are housed in a frame constructed from slotted steel angle iron and aluminum plates as shown in Figure 1. Labtech Notebook software has been configured for controlling the multiple heater array, fiber translation mechanism, fiber feed and fiber take-up mechanism. The fibers were laser impacted with a laser beam diameter of 2.5mm while translating through the laser melt zone. The processed fibers were characterized at the surface, as well as at the cross-section, using secondary electron images on a Phillips Model 505 SEM. The elemental distribution and interdiffusion were determined using Philips Model PV 99W EDS energy dispersive X-ray analyzer. The fibers were tested for critical temperature using flux exclusion technique and critical current densities using a four probe technique.

III. RESULTS AND DISCUSSION

Initially, extruded fibers of 123 materials were investigated (1). The fragile characteristic of the processed fiber resulted in handling difficulty, therefore, core coated high T_c superconducting fibers were identified as a more promising candidate for LDT process. Duranickel 301 (95%Ni • 4.4%Al • 0.6%Ti) core coated high T_c superconducting Dy-Ba-Cu-O fibers obtained from Pacific Superconductors were translated in the thermal gradient. Work was discontinued on the Duranickel 301 core coated high T_c superconducting Dy-Ba-Cu-O fibers due to the interdiffusion of copper (Cu) and nickel (Ni) between the coating and the core at 800°C, which is below the peritectic melting temperature of high T_c superconducting Y-Ba-Cu-O materials, thereby resulting in destroying the superconducting property of the fiber (2-4).

Work was performed on yttria stabilized zirconia core coated high T_c superconducting Y-Ba-Cu-O fibers obtained from ICI superconductors. Figure 3 shows the secondary electron image surface morphology of the yttria stabilized zirconia core coated high T_c superconducting Y-Ba-Cu-O fibers along the fiber-axis direction. Figure 3(a) shows the surface morphology of an unprocessed fiber. Figure 3(b) shows the surface morphology of a fiber translated in the thermal gradient at a rate of 1.7cm/hr, in the presence of 42.6ml/min flowing oxygen, with the laser melt zone temperature at 1080°C. As seen from the micrograph elongated textured grains greater than 10x60μm dimensions were observed to extend in the direction of translation which is along the axis of the fiber. A high degree of crystal continuity and alignment was observed at the boundary region. Figure 4 shows the cross-sectional view of the yttria stabilized zirconia core coated high T_c superconducting Y-Ba-Cu-O fiber. Figure 4(a) shows the secondary electron image of an unprocessed fiber. The core diameter is observed to be 810μm, whereas the coating thickness is observed to be 50μm. Figure 4(b) and 4(c) shows the typical energy dispersive X-ray (EDX) map of the elemental distribution of barium (Ba) and yttria (Y) over the fiber cross-section when processed under the parameters of Figure 3(b). No elemental interdiffusion was observed between the coating and the core for all the elements under consideration.

Figure 5 shows the secondary electron image surface morphology along the fiber axis direction of the yttria stabilized zirconia core coated high T_c superconducting Y-Ba-Cu-O fibers translating at a rate of 1.7cm/hr in the thermal gradient, in the presence of 42.6 ml/min flowing oxygen, with the laser melt zone temperature at 1020°C. Figure 5(a) shows the microstructure of a non-laser impacted zone of the fiber translated in the thermal gradient, whereas Figure 5(b) shows the surface morphology of the fiber impacted with a laser radiance of $49W/cm^2$ while translating at a rate of 1.7 cm/hr in the thermal gradient with the laser melt zone temperature at 1020°C. By comparing Figure 3(b) and 5(b) it can be observed that the laser-driven directional solidification in a controlled thermal gradient has a higher growth rate and a lower thermal budget than the conventional thermal gradient processing. Textured elongated grains in the fiber axis direction can be observed at 1020°C as compared to 1080°C in the conventional thermal gradient. Besides having higher growth rates the significance of laser heating is that complete melting to the single phase melt occurs in 53sec, as compared to 10 hours by resistively heating, a time scale too short to allow significant segregation under the driving force of the peritectic reaction. Figure 6 shows the final thermal profiles developed from the results obtained and are compared between resistively heated and laser heated to the single phase melt zone. Further considering the 53sec time scale in the incongruent melt region the intra-grain inclusions of Y_2BaCuO_5 will also be smaller so as to be beneficial for flux pinning thereby improving the critical current properties of the high T_c superconducting polycrystalline fibers/wires for device applications. A superconducting critical temperature was observed to be 80K in the processed fiber as compared with 95K in the unprocessed yttria stabilized zirconia core coated high T_c superconducting Y-Ba-Cu-O fibers. A critical current density of $1.8x10^2$ $Amps/cm^2$ at 77K, 0.6T was observed in the processed fiber as compared to $2.8x10^2$Amps/cm^2 in the unprocessed fibers as shown in Figure 7. The results indicate that careful control of the specimen processing can lead to the formation of optimized samples which are highly densified and textured due to recrystallization following melting of the material in the single phase melt region. Work is in progress to further optimize the process parameters in order to improve the critical current density of the processed fiber.

IV. CONCLUSIONS

Based on the analytical discussion and experimental results, it can be concluded that the laser-driven directional solidification in a thermal gradient technique is highly feasible for development as a continuous process for improving the critical current densities of high T_c superconducting polycrystalline fibers/wires with elongated textured grains. The time spent in the incongruent melt region, on the order of 53sec, is too short as compared to 10 hours in a conventional thermal gradient to allow significant segregation under the driving force of the peritectic reaction. Also the possibility of Y_2BaCuO_5 inclusions is much smaller, which is beneficial for flux pinning. The technique has a significant advantage of yielding higher growth rates and lowering the thermal budget as compared to the conventional thermal gradient, and further suppressing weak-link behavior by orientation of crystals, formation of dense structures with enhanced connectivity, formation of fewer and cleaner grain boundaries and minimization of phase segregation in the incongruent melt region, thereby enhancing J_c in the high T_c superconducting polycrystalline fibers/wires. The technique is capable of fabricating polycrystalline fibers with high density, mechanical strength and critical current density and is amenable to scale up.

ACKNOWLEDGMENTS

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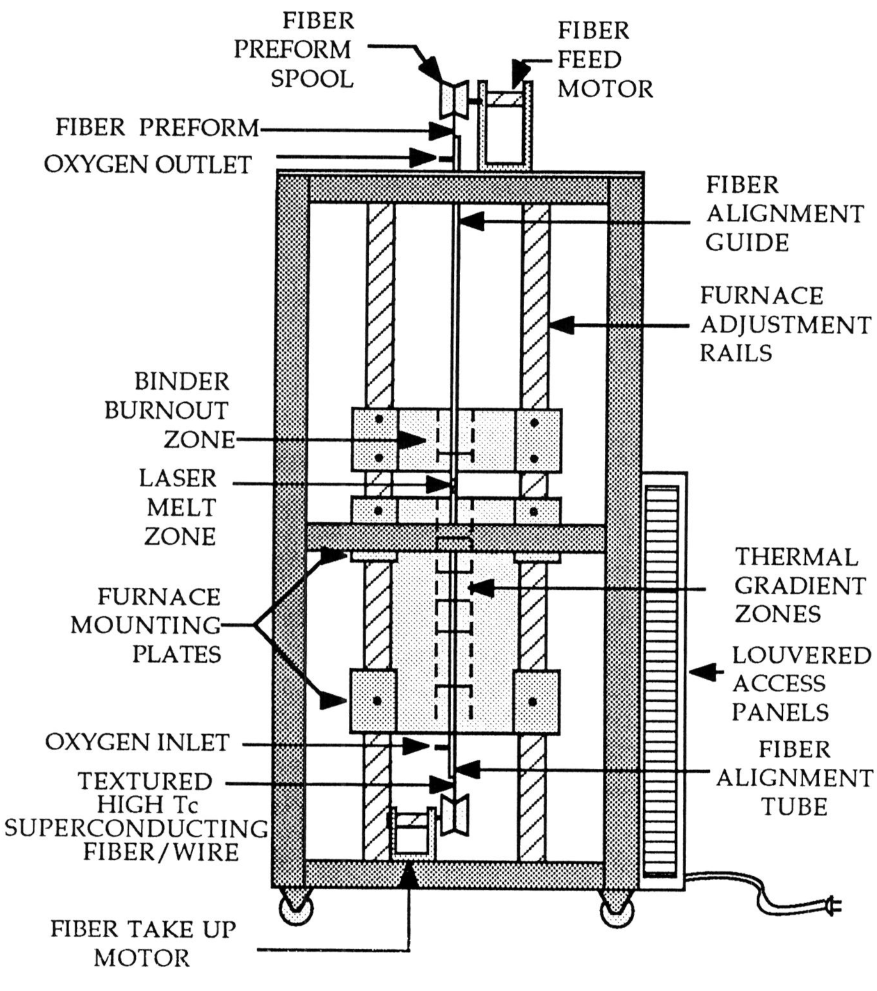


Figure 1. Thermal gradient furnace comprised of a multiple heater array.

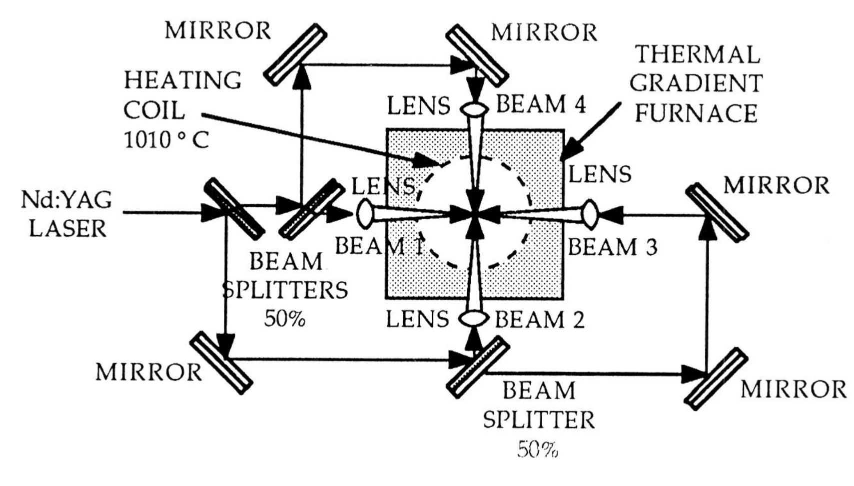
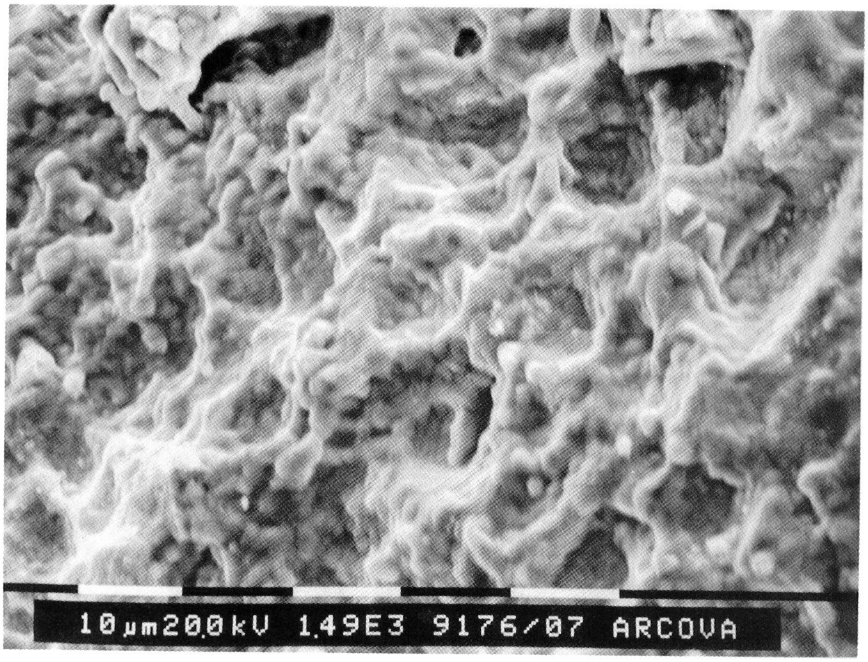
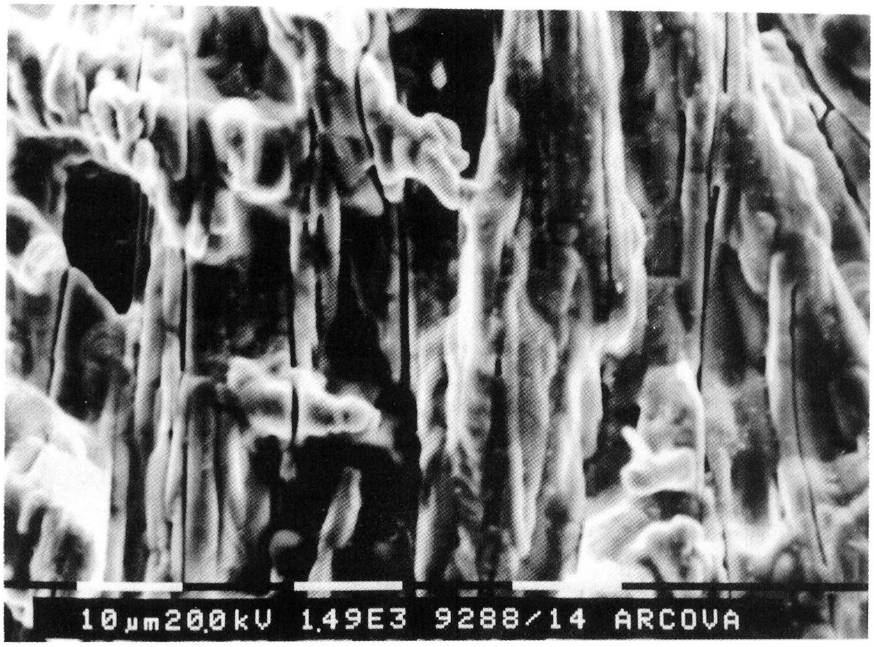


Figure 2. Top view of the laser optics in the laser melt zone of the thermal gradient furnace.



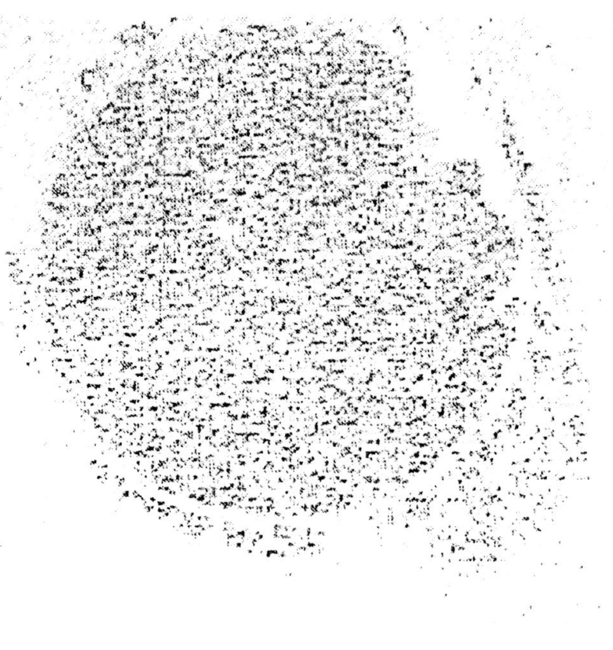
(a) Unprocessed



(b) Translated in the Thermal Gradient at a Rate of 1.7 cm/hr, in the Presence of 42.6 ml/min Flowing Oxygen, with theLaser Melt Zone Temperature at 1080°C.

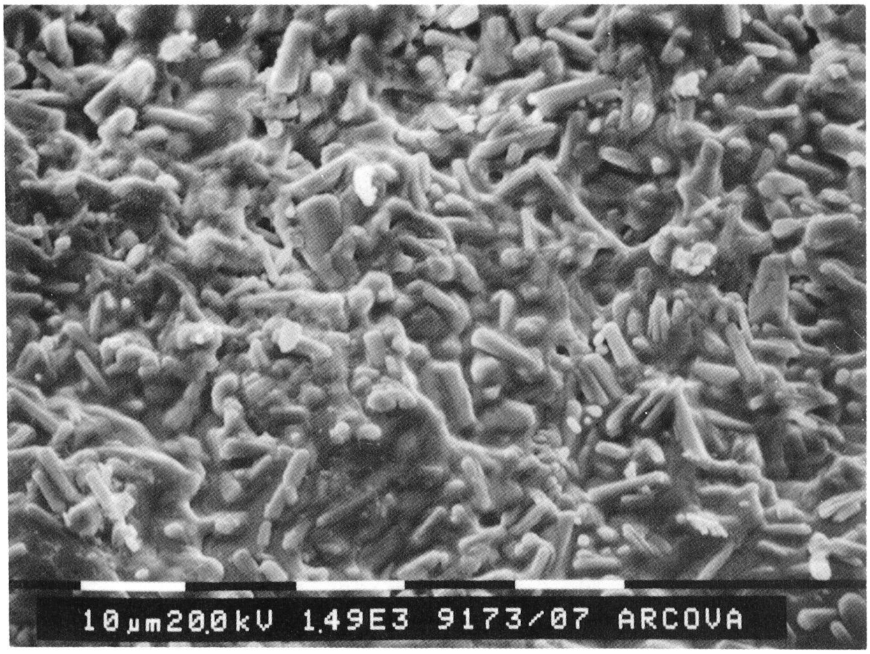
Figure 3. Secondary electron image surface morphology along the fiber axis direction of the yttria stabilized zirconia core coated high T_c superconducting Y-Ba-Cu-O fibers.

(a) Secondary Electron Image of an Unprocessed Fiber

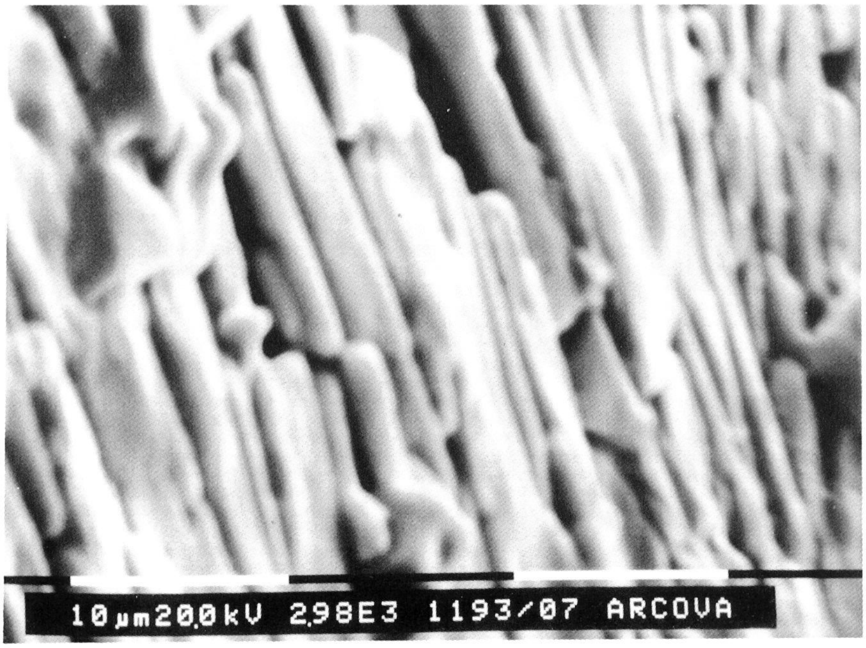


(b) X-ray Map of Ba in the Processed Fiber (c) X-ray Map of Y in the Processed Fiber

Figure 4. Cross-sectional view of the yttria stabilized zirconia core coated high T_c superconducting Y-Ba-Cu-O fibers.



(a) Non-Laser Impacted



(b) Laser Impacted with 49 W/cm^2 Radiance while Translating

Figure 5. Secondary electron image surface morphology along the fiber axis direction of the yttria stabilized zirconia core coated high T_c superconducting Y-Ba-Cu-O fibers translating at a rate of 1.7cm/hr in the thermal gradient, with the laser melt zone temperature at 1020°C.

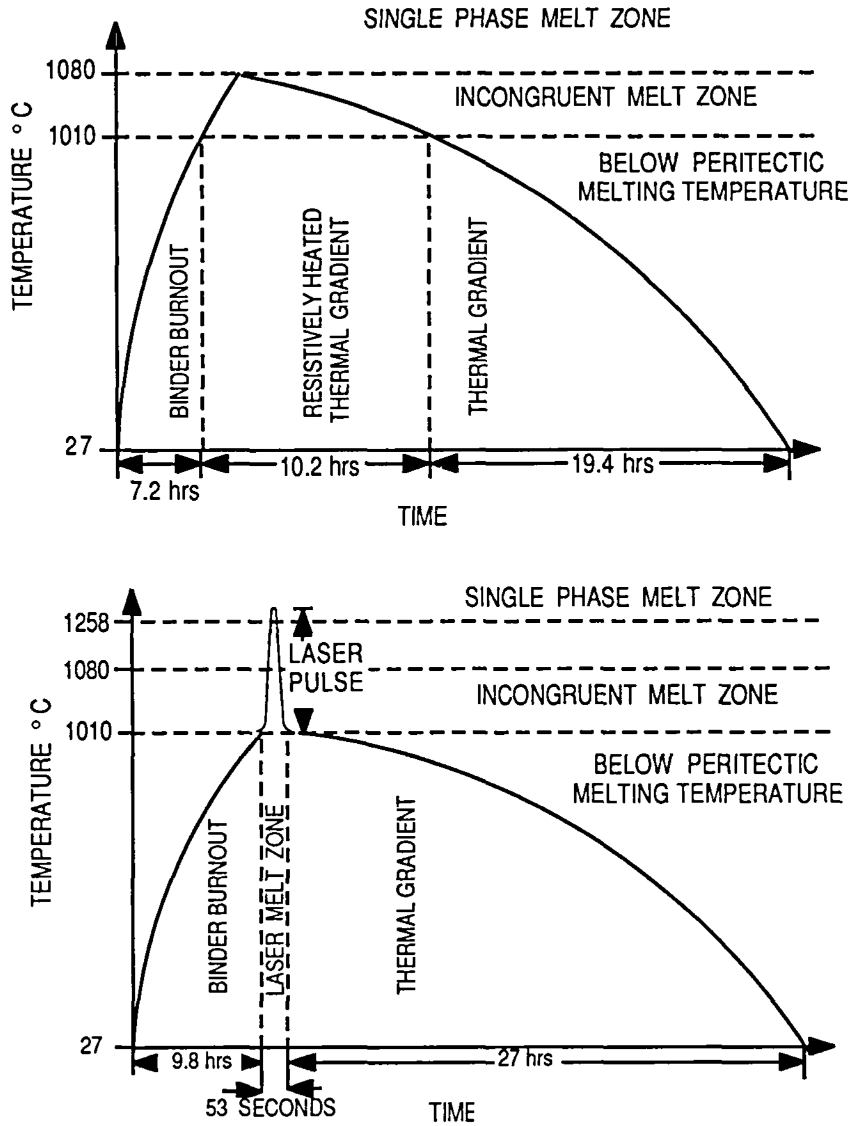


Figure 6. Thermal profiles of fibers heated to a single phase melt zone and translated in a thermal gradient for continuous processing (not to scale).

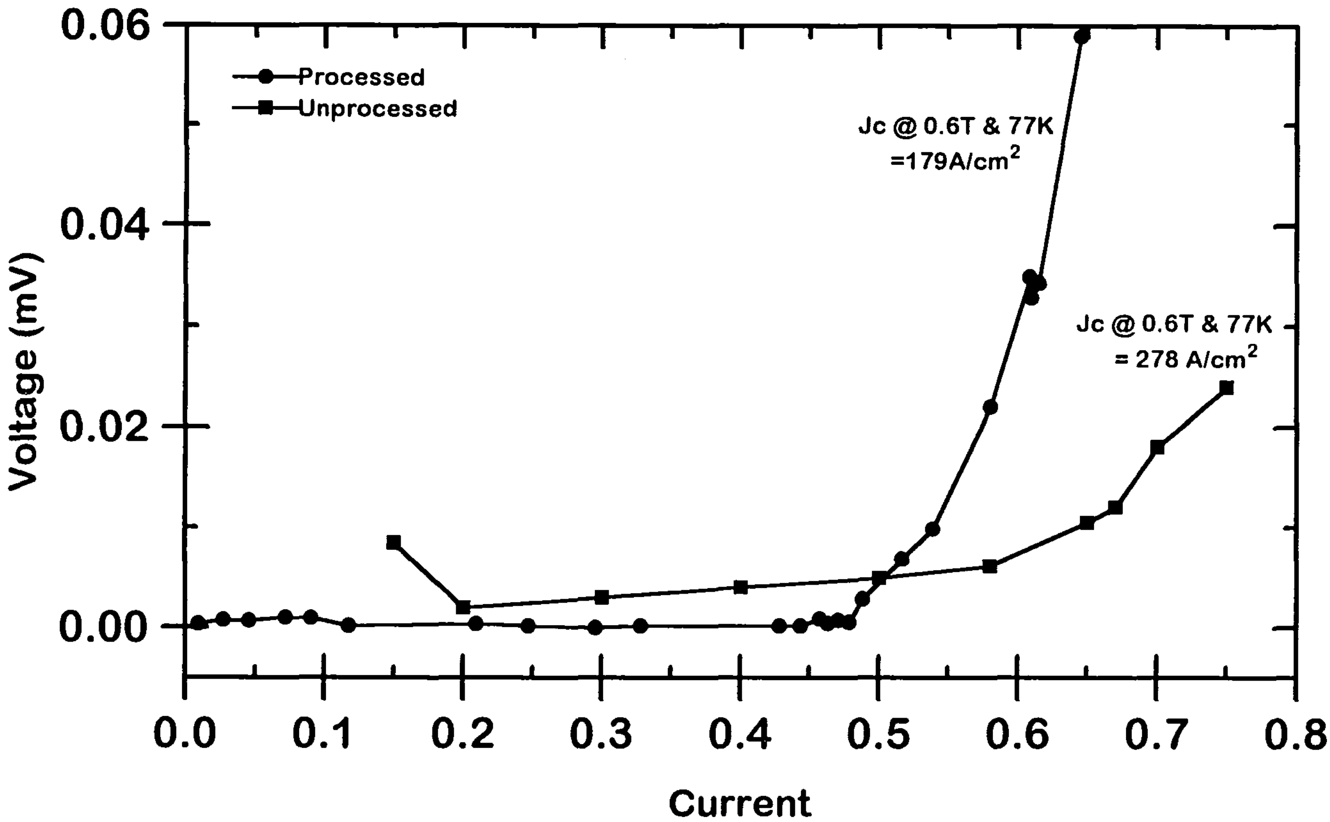


Figure 7. Critical current densities of the yttria stabilized zirconia core coated high Tc superconducting Y-Ba-Cu-O fibers.