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Processing Yttrium-Barium-Copper Oxide Superconductor Zero Gravity Using a Double Float Zone Surface

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DISCLAIMER

PROCESSING YTTRIUM-BARIUM-COPPER OXIDE SUPERCONDUCTOR ZERO GRAVITY USING A DOUBLE FLOAT ZONE FURNACE

by

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ABSTRACT

The effects of processing $YBa_2Cu_3O_x$ (Y123) superconductor in the near-zero gravity (0g) environment provided by the NASA KC-135 airplane flying on parabolic trajectories were studied. A new sheet float zone furnace, designed for this study, enabled fast temperature ramps. Up to an 18-gram sample was processed with each parabola. Samples of Y123 were processed as bulk sheets, composites containing Ag and Pd, and films deposited on single crystal Si and MgO substrates. The 0g-processed samples were multi-phase yet retained a localized Y123 stoichiometry where a single ground-based (1g) oxygen anneal at temperatures of 800°C recovered nearly 100-volume percent superconducting Y123. The 1g processed control samples remained multi-phase after the same ground-based anneal with less than 45 volume percent as superconducting Y123. The superconducting transition temperature was 91 K for both 0g and 1g processed samples. A 29 wt.% Ag/Y123 composite had a transition temperature of 93 K. Melt texturing of bulk Y123 in 0g produced aligned grains about a factor of three larger than in analogous 1g samples. Transport critical current densities were at or below 18 A/cm^2, due to the formation of cracks caused by the rapid heating rates required by the short time at 0g. Y123 deposited on single crystal Si and MgO in 0g was 30 vol.% Y123 without an anneal. A weak superconducting transition at 80 K on MgO showed that substrate interactions occurred.

Introduction

A near-zero gravity environment (henceforth referred to as zero gravity or 0g) presents unique opportunities for processing materials of scientific interest. Samples processed under 0g conditions differ from those processed under gravity-one (1-g) conditions primarily in their microstructure due to the absence of gravitational driven buoyancy, sedimentation, buoyancy-driven convection, and hydrostatic pressure. The absence of gravitational force also allows for a variety of container-less processing strategies where a material can be processed free from contact with pre-existing solids. General reviews of materials processing in 0g are offered by Walter[1], Doremus and Nordine[2], Nauman and Herring[3], and Avduyevsky[4].

Materials whose useful properties depend on their microstructure are prime candidates for processing in 0g. Materials that are highly reactive or have a tendency to form undesired phases from contact with pre-existing solids are likewise candidates. The high-temperature superconducting ceramics [i.e., $YBa_2Cu_3O_x$ (Y123)] meet both of these criteria[5,6].

Processing Y123 near the melting point (1025°C) has many positive effects such as grain growth/ connection, densification, and texture development[7]. Melting under 1g conditions, however, can be

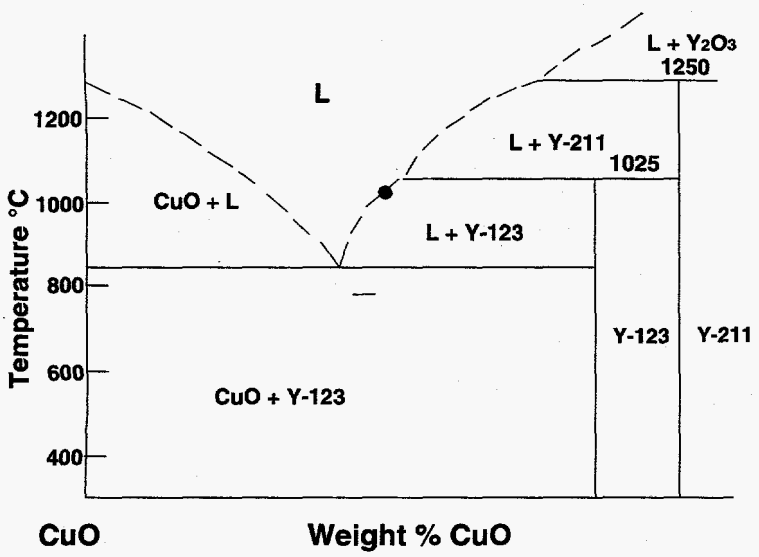

Figure 1: Pseudo-binary phase diagram in schematic form with temperature (°C) versus weight % CuO for the Y123-CuO system.

detrimental to the formation of a single phase homogeneous superconductor. These effects arise in part from the fact that Y123 undergoes a peritectic dissociation upon melting, forming a liquid and solid of different densities that can separate due to sedimentation forces present at 1g conditions[8]. This incongruent melting of Y123 is illustrated in a simplified pseudo-binary phase diagram[9] shown in Fig. 1. At a Y123 composition, exceeding 1025°C results in an equilibrium between $BaCuO_2$-rich liquid and Y_2BaCuO_5 (Y211) solid. Upon cooling of the Y123 sample, Y211 grains are left in a $BaCuO_2$-rich matrix. These can be partially back-reacted to form the Y123 superconductor upon annealing in excess of 900°C for tens to hundreds of hours. However, when solid state diffusion/reaction dominates, as in the anneal, previous phase segregation causing localized off-stoichiometry results in the inability to recover the Y123 composition. Normally, this effect is minimized during ground-based superconductor synthesis by avoiding temperatures where free-flowing liquid phases are present or by quench cooling where time is insufficient for extensive phase separation while in the liquid state. In spite of following these procedures in 1g, multiple sintering steps with intermediate grindings are needed to produce single- phase material. In addition, it has been established that container materials will react with Y123 composition liquid resulting in degradation of the superconducting properties[8].

Zero gravity offers a processing strategy where Y123 can be brought to a temperature above which free-flowing liquid phases are present and not suffer from phase separation. In addition, 0g can provide an environment where contact with wall materials that offer unwanted chemical reactions can be avoided.

A series of experiments on Y123 processing have been completed using the near 0g conditions provided by the NASA KC-135 airplane flying aeroparabolas. A furnace with the sample in the form of a sheet [Sheet Float Zone (SFZ) furnace] creates a float zone as a line across the sample and was developed for this study. The duration of 0g offered by aeroparabolas is sufficient for the melting and solidification of Y123, but not for the annealing. Since the resulting multi-phase solid has no gross-phase separation, a subsequent 1-g anneal at a temperature below which free flowing liquids are present allows reformation of single phase Y123 that retains effects of the original 0g processing.

Experimental

Furnace Operation: The SFZ furnace used in these 0g studies was developed in a collaboration between Space Industries and Los Alamos National Laboratory. A detailed description of this furnace will be published[10]. The furnace control and data acquisition were performed with a Macintosh II computer using a LabView application (National Instruments). Four quartz halogen heaters (1000 watts each) with linear elliptical reflectors heated the sample. A scanning optical pyrometer (5 μm wavelength) was used to determine the temperature (+-50°C) vertically across the center of the sample (4-mm-wide scan). The pyrometer was calibrated in 1g with Y123 test samples containing embedded thermocouples. Sample processing temperatures, ranging from 950-1300°C, were recorded at a rate of 4 scans per second.

The operating principles are illustrated in Fig. 2. Each sample sheet, held by its horizontal edges in a frame, was positioned in the furnace and continuously purged with air (<50ppm CO_2). The quartz heaters projected a focal line (63-mm long) horizontally across the sample center (56-mm maximum sample width) where the upper and lower pairs moved vertically apart to enable float zone melting over the central region (15-48 mm). Rapid scrolling of the heaters up and down preheated samples to about 800°C prior to entering 0g. Heating rates of 40 and 100°C/s were used to heat the sample from 800°C to the desired processing temperature (1100-1300°C) upon entering 0g. In 0g, a molten zone (5-mm high) was established and split into two molten lines moving in opposite directions as the heaters moved apart (vertically) at scroll rates of 0.63-2.0 mm/s. The solidifying area between these molten zones is supported by liquid bridges and, therefore, is not in direct contact with the original solid sample or any container walls. An alternate operating procedure was to hold the heaters stationary and form a stationary molten line for the duration of the 0g.

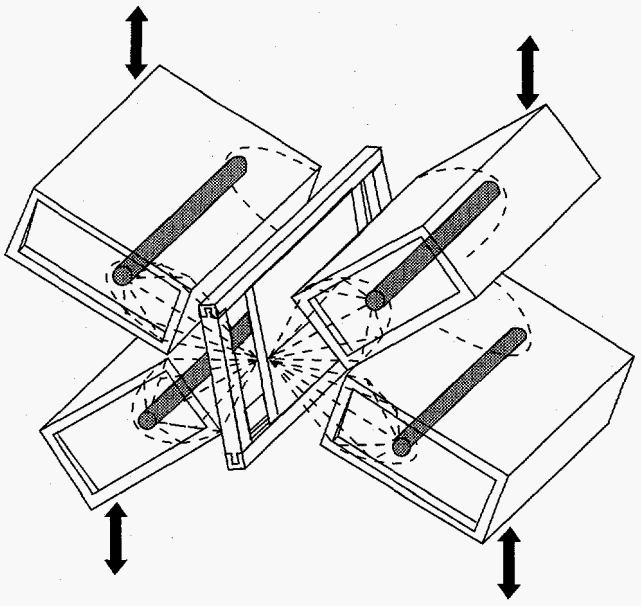

Figure 2: Schematic showing the sample geometry in relation to the heaters for the Sheet Float Zone furnace.

Zero-gravity conditions for these experiments were produced on the NASA KC-135 airplane flying on a parabolic trajectory. The KC-135 gives cyclic periods of about 0.01 g for about 18 seconds each, followed by a period of 1.8 g for about 50 seconds. Up to 40 samples were run each flight. This report covers results from a total of 25 separate flights made over the period from August 1988 to March 1991.

Control samples were terrestrially processed at 1g in the SFZ furnace under conditions analogous to those used in their 0g counterparts. These control samples were never intended for comparison to those of the best 1-g processed superconductors and served only to address the effects of processing Y123 under conditions advantageous for 0g. The control samples did require a modified process to prevent the molten sample from falling out of the holder by limiting the (vertical) heater movement to less than 10 mm or by holding the heaters stationary. There was significant slumping of all samples studied under 1-g conditions.

The short times associated with SFZ processing necessitated post-flight sample annealings in a conventional furnace under 1-g conditions. This treatment was accomplished by heating the samples in flowing O_2 at 800°C for 10 hours followed by 450°C for 10 hours. These annealing temperatures were chosen so not to create phases that would separate under the influence of gravity and erase the structure imparted by 0g processing while trying to optimize the superconducting properties.

Sample Preparation: Samples of Y123 examined in this study were synthesized from milled stoichiometric amounts of Y_2O_3, $BaCO_3$, and CuO powders. The mixtures were cold pressed into sheets (75x25 mm) with thicknesses varying from 1-4 mm. The samples were held by the 25-mm-wide edges for SFZ processing. These sheets were air sintered twice at 920°C for periods of 24 hours and had a density of 85% of theoretical. Films of Y123 were deposited on substrates of single crystal Si or MgO by using a methanol slurry followed by air drying.

Composites were prepared from ball-milled presintered Y123 powder with Ag or Pd powders (1-3 μm diameter) to achieve 29 wt.% dopant concentrations. Other sheet samples contained a $Y_2Cu_2O_5$ and $4BaCuO_2$ mixture[11] to directly synthesize Y123 under 0g conditions. Copper-rich samples with a $YBa_2Cu_{4.4}O_x$ composition commercially obtained from Hi Tc SuperConco Inc. were used to determine if copper loss occurred.

Sample Characterization: X-ray diffraction data were obtained using a Scintag XDS-2000 diffractometer on samples both before and after processing in the SFZ furnace. X-ray analyses were performed on pieces removed from the central portion of the samples to avoid edge effects. Diffraction lines were analyzed by correlating with known patterns established for the phases of interest.

Sample microstructures were examined by optical reflectance microscopy and scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) using a CAMSCAN SEM with a Kevex X-ray detector and Quantex software V package.

The volume magnetic susceptibility, χ, was measured with a Quantum SPMS at a 100-gauss applied field. The theoretical low-field value for an ideal superconductor gives a value of $\chi = -1/4\pi$; thus,the quantity $4\pi\chi$ provides a convenient measure of the sample volume fraction that is superconducting. Transport critical currents were determined as a function of temperature and applied magnetic fields by passing a current through the sample while measuring the voltage drop in a four-point contact measurement.

Results

Table I summarizes the processing conditions for all the Y123 samples.

Table I
Summary of Y123 Samples Processed with the SFZ Furnace
in Zero Gravity from 8/88 to 3/91

Sample Type	Number of Samples Processed in 0g	Scroll Rate† (mm/s)	Approximate Heating Rate (°C/sec)	Estimated Max. Temp (°C)
Y123 Bulk:				
orthorhombic	45	1-2	40-100	1100
	22	0.63	100	1200
	18	0	100	1200
tetragonal	30	1-2	40-100	1100
	19	0.63	100	1200
	9	0	100	1200
$Y_2Cu_2O_5$/				
$4BaCuO_2$	31	1-1.5	40-100	1100
	18	0.63	100	1200
	4	0	100	1200
$YBa_2Cu_{4.4}O_X$	17	1-1.5	40-100	1100
	4	0	100	1200
Y123/:				
29wt.%Ag	12	0.75-1	40-100	1200
	11	0	40-100	1200
29wt.%Pd	5	0.75-1	100	1200
	4	0	100	1200
Y123 film:	28	0.63-1.25	100	1300
Single Crystal Si				
Single Crystal MgO	17	0.63-1.25	100	1300

† 0 mm/s when heaters were held in place and melted a stationary line in sample.

Initial Sample Synthesis: It was demonstrated that Y123 sample preparation in 1g by a conventional cold pressing and sintering of oxide/carbonate mixtures produced bulk sheet samples, which were single phase (from X-ray) with a superconducting transition of 92 K, and a superconducting volume fraction of essentially 100% by 77K.

Bulk Y123 Processing: About 5 to 18 grams from the center of the Y123 sheets were processed with each parabola, depending on the scroll rate. Processing in the SFZ furnace of Y123 samples with a bulk density of 60% of theoretical led to significant sample shrinkage on melting that contributed to instability of the molten zone. Samples with densities of 85% of theoretical or higher did not usually have this problem and were used for the balance of this study. There were some undesirable effects noted at the free edges of the molten zone during processing. The edges radiatively cooled faster than the middle, resulting in horizontal temperature gradients. Sometimes the center would be molten while the edges remained slushy. The slushy edges undoubtedly provided some stabilizing force for the tendencies of the edges to neck inwards. The denser samples required a preheat to prevent fracture during the high heat fluxes attained upon melting. Sample fracture during cooling was a problem and was reduced by mounting one end of the sample so that it could move upon thermal expansion and contraction.

It was found that SFZ processing of Y123 samples in the fully oxygenated orthorhombic state formed bubbles in the molten zone that left the solidified samples highly vesicular due to the liberation of oxygen during heating. Beginning with tetragonal samples in a lower oxygen state ($YBa_2Cu_3O_{6.2}$) formed the fewest voids and resulted in the densest structure.

The microstructure of a tetragonal Y123 processed in the SFZ furnace under 0g at 1100°C with a scroll rate of 1.25 mm/s is shown in Fig. 3a (unannealed). The sample is multi-phase and fine-grained. Fig. 3b shows an adjacent piece of the same sample after annealing. The multi-phase nature of the unannealed sample is converted into nearly single phase, interlocking grains of Y123. An X-ray diffraction spectrum of a sample processed under similar conditions to that in Fig. 3a is shown in Fig. 4a. The material is multi-phase containing Y_2BaCuO_5, $BaCuO_2$, and CuO with very little Y123 remaining. After an anneal, the X-ray diffraction spectrum shows nearly single phase Y123 as seen in Fig. 4b. Zero-g processing, while resulting in a multi-phase solid, preserves the localized Y123 stoichiometry so that a single ground-based anneal can recover nearly single phase Y123. If phase separation occurred, the localized stoichiometry would be off of the Y123 composition, and a single ground-based anneal would be insufficient to recover Y123. Analogous results were also obtained with the $Y_2Cu_2O_5/4BaCuO_2$ samples; thus, there were no advantages found to directly synthesize Y123 in 0g.

The microstructure and X-ray spectra for the unannealed 1-g processed samples are not distinctively different from the unannealed 0g samples. After annealing, however, a large difference is observed as seen in Fig. 5 (microstructure) and Fig. 6 (X-ray). The samples remain multi-phase with only a fraction as Y123. The unannealed multi-phase structure for the 1-g processed sample is not dissimilar to that for the unannealed 0g sample; yet phase separation in 1g causes non-uniformity in the localized stoichiometry where a single ground-based anneal cannot recover the Y123 composition and is apparent by comparison of Figs. 5 to 3b and Fig. 6 to 4b.

Annealed copper-rich $YBa_2Cu_{4.4}O_X$ samples showed significant $BaCuO_2$ and CuO present following processing in both 0g and 1g; thus there was minimal loss of copper during processing, and the Y123 stoichiometry proved best.

The samples processed in 0g and annealed had superconducting transition temperatures of 91 K and $4\pi\chi$ values at 7 K approaching unity. Equivalent samples that had been totally ground-based processed had a transition temperature of 91 K and $4\pi\chi$ values at 7 K of less than 0.45. The temperature dependence of the magnetic susceptibilities for the 0g and 1g samples is plotted in Fig. 7, where at 77 K, the 0-g and 1-g samples have $4\pi\chi$ values of 0.32 and 0.13, respectively.

It was possible to achieve melt texturing in both bulk- and thin-film Y123 samples processed with the SFZ furnace under 0-g and 1-g conditions. Y123 samples became melt-textured when subjected to

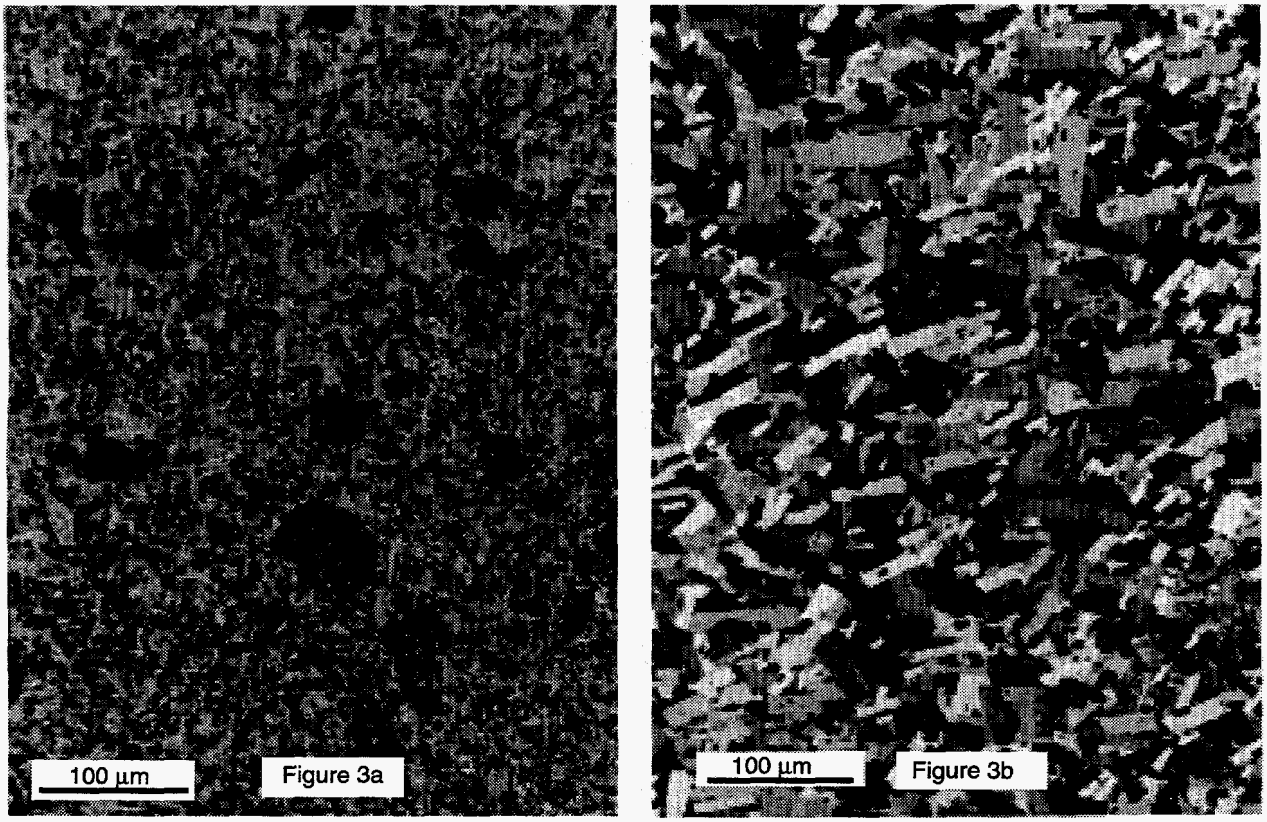

Figure 3: Microstructure in 0g processed bulk Y123: a) unannealed and b) after a 1g anneal.

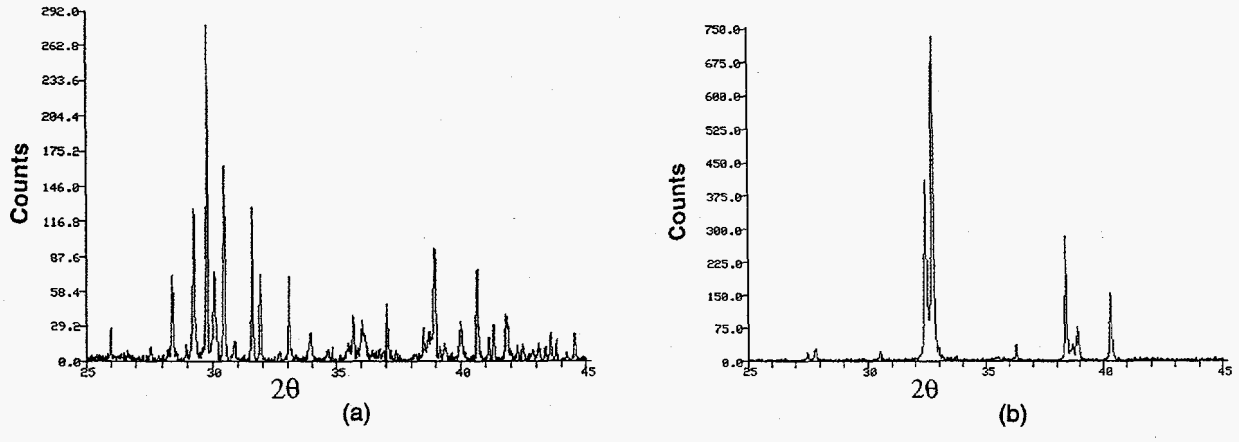

Figure 4: X ray diffraction spectrum given for 0g processed Y123 material: a) unannealed, b) after a 1g anneal.

1200°C or greater with a scroll rate of 0.63 mm/s and are shown in Figures 8 and 9, respectively. Samples processed under 0g have grains 100-200 µm in width and 1- to 2-mm long, whereas samples subjected to an analogous treatment under 1g have grains 30-120 µm in width and 0.8 to 1.4-mm long. Caution must be exercised in conclusions drawn about the role of gravity in causing this difference since the processing conditions for the 0g case cannot be exactly duplicated in 1g. Transport critical current densities of the samples processed under 0g were low, with a best value of 18 A/cm^2. Critical current measurements were not conducted on the samples processed under 1g.

Fig. 10 shows a cracking phenomena observed in a few Y123 samples. Proceeding across the usual melt texture grains are a series of periodic cracks with a spacing of 200 μm, which has developed parallel to the molten zone. The melt textured grains were shown by EDS to have a Y123 composition, whereas each crack was filled with a frozen liquid having a composition rich in $BaCuO_2$. If this were to happen to some degree in all the melt-textured samples (those in Fig. 10 being an extreme), then the presence of these cracks and insulating phases may explain the low values of critical current. It is believed that these cracks arise from the rapid heating rates required by the short time at 0g.

Composite Samples: Composite samples made with silver were chosen because of the interest in providing fine, disperse sites for flux pinning. Silver (961°C melting point) melts before the Y123 matrix (1025°C melting point) and forms agglomerates due to gravitational and capillary forces. Palladium composite samples in concentrations similar to ours are not necessarily detrimental to the superconductivity[12]. We chose the palladium (1552°C melting point) for a microstructural comparison to the silver where the immiscible phase now has a melting point greater than that of the Y123.

Composites of Ag/Y123 displayed behavior in 0g dependent on the rate of heating. Silver pooling on the outer free surface was observed in the 29 wt% Ag/Y123 samples and a heating rate of 40°C/s as shown in Fig. 11. In spite of the pooling, much of the silver remained as finely dispersed grains (3-55 μm diameter). With the 100°C/s heating rate, the silver remained dispersed in the melt (3-25 μm diameter) and no observed pooling as seen in Fig. 12. There was no coarsening of the silver grains as a result of the anneal. The temperature dependence of the magnetic susceptibility for the Ag/Y123 composite processed under 0g is shown in Fig. 7. The Ag/Y123 sample has a transition temperature of 93 K and values of $4\pi c$ (corrected for the volume fraction of silver) at 7 and 77 K of 0.95 and 0.67 respectively. Compared to the bulk Y123 samples processed in 0g and 1g, the Ag/Y123 sample shows a sharper change in

Figure 5: Microstructure in 1g processed, 1g annealed Y123. The unannealed 1-g processed samples were similar in appearance to that of the unannealed 0g samples as shown in Fig. 3a.

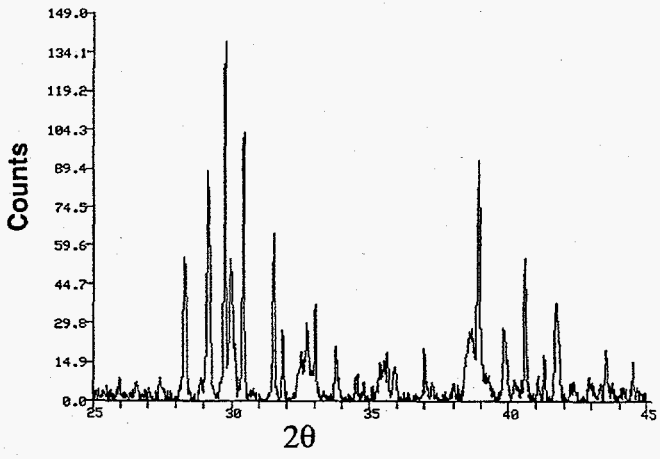

Figure 6: X-ray diffraction spectrum given for 1-g processed, 1-g annealed Y123. The unannealed 1-g processed samples were similar to that of the unannealed 0g samples as shown in Fig. 4a.

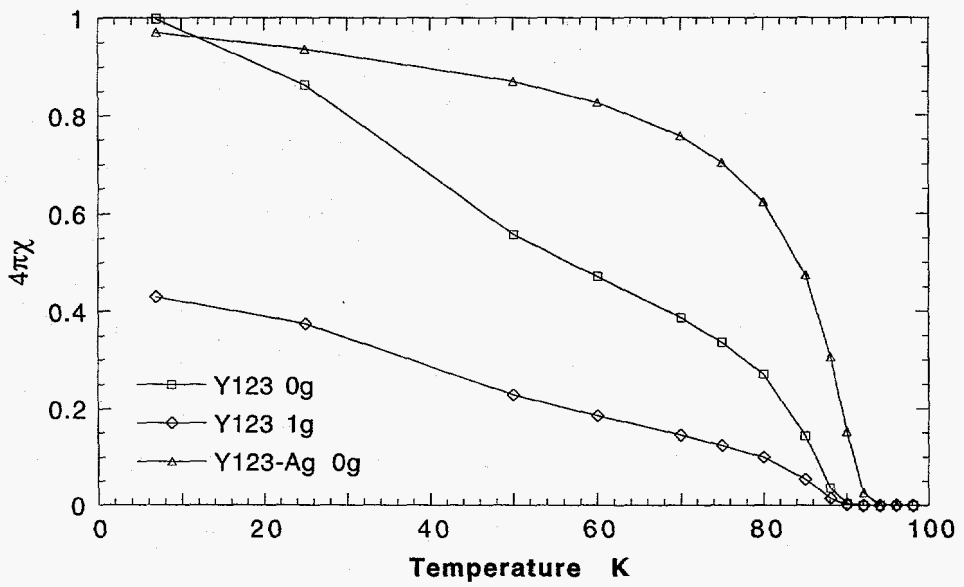

Figure 7: $4\pi\chi$ versus temperature (K) at 100 gauss applied field for samples of Y123 processed in 0g, 1g, and of a 29 wt% Ag/Y123 composite processed in 0g.

the susceptibility as the transition temperature is approached.

Processing the silver composite under 1-g conditions resulted in silver pooling for all heating rates, which essentially stripped the fine diameter silver from the bulk of the Y123 as shown in Fig.13 for an Ag/Y123 sample. Silver pools and agglomerates 60 μm to several millimeters in diameter are formed. There was no coarsening of the silver grains as a result of the anneal. The transition temperature and values of $4\pi c$ were similar to the bulk Y123 processed in 1g.

Processing of the 29 wt.% Pd/Y123 composites under 0g and the faster heating rate resulted in non-spherical Pd agglomerates (3-25 μm in characteristic length) in spite of temperatures remaining 350°C below its melting point. There was no coarsening of the palladium grains as a result of the anneal. Electrical measurements were not made on these samples.

<u>Y123 Film Processing:</u> The Y123 films (unprocessed thickness of 100-200 μm) deposited on single crystal Si and MgO substrates were preheated to about 800°C and then rapidly heated to 1300°C while the heaters scrolled across the sample (0.63-1.25 mm/s). The molten zone formed a free-flowing liquid that freely wetted the surface. Maringoni convection was observed at the leading

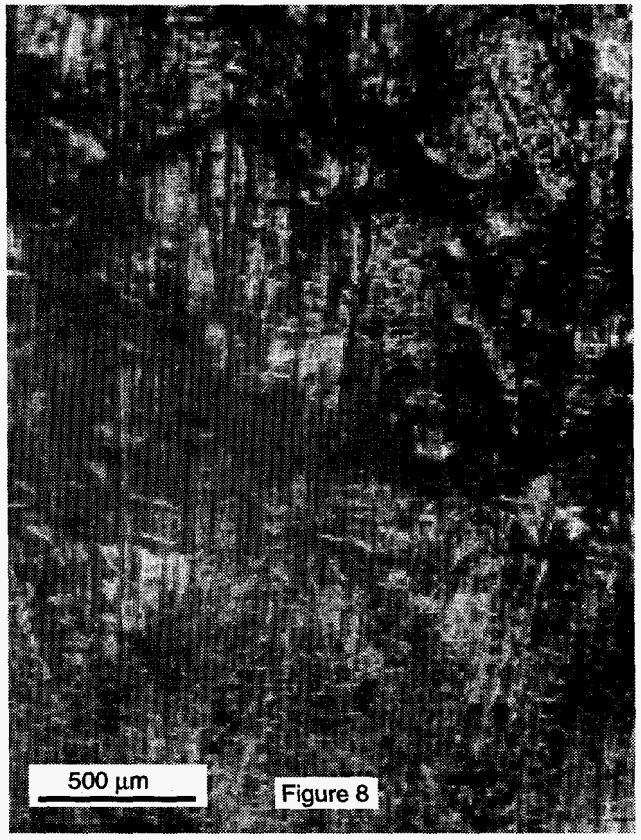

Figure 8: Microstructure in 0g processed, melt-textured, Y123 with vertical orientation in direction of zone movement.

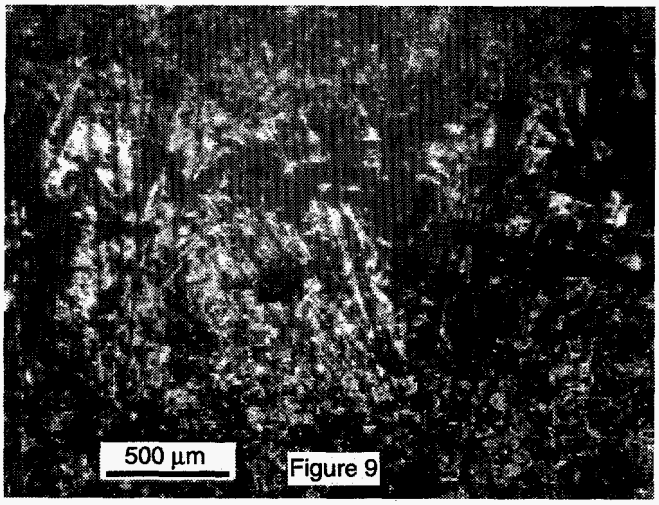

Figure 9: Microstructure in 1-g processed, melt-textured, Y123 with vertical orientation in direction of zone movement.

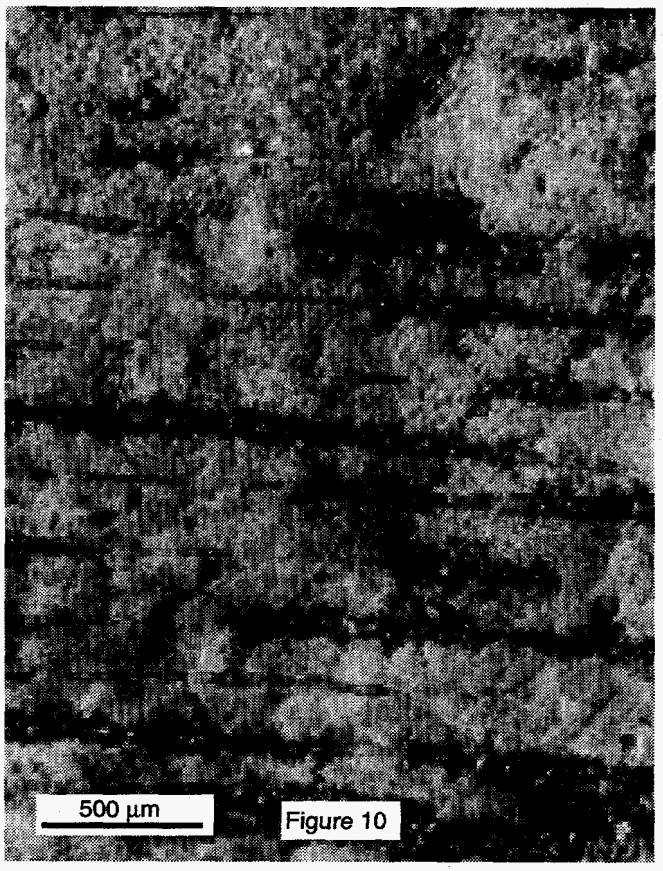

Figure 10: Periodic cracking structure in the unpolished surface of 0g-processed, melt-textured, Y123 with vertical orientation in direction of zone movement.

edge but not at the trailing edge. There were no visible signs of gas evolution, and the resulting layers were dense and bonded to the substrates. The Y123 films were not annealed. The unannealed microstructure of the films showed interlocking grains of Y123 (5-10-µm-thick layer) next to the Si or MgO with an outside covering (10-20-µm-thick layer) of a $BaCuO_2$ rich phase. About 30 vol.% of the unannealed film was Y123, determined from optical inspection. X-ray diffraction analysis showed the presence of Y123 as illustrated for a Y123 film on Si in Fig. 14. However, the superconducting transition was not discernible for the Y123 film on Si and, on the MgO, was weak and lowered to 80K with a transport critical current of about 0.1 A/cm^2, indicative of substantial substrate interactions in spite of the short processing time. Substrate cracking due to thermal shock immediately following processing was common.

Discussion

This work showed that we were not able to fabricate the yttrium-based high-temperature superconductor under 0g conditions with electrical properties as good as what can be achieved from ground-based fabrication. However, we chose to process samples under 0g conditions that took full advantage of that environment; thus, the ground-based controls were not necessarily good in comparison to the best possible ground-based samples and served only to compare the effects of the reduced gravity processing.

We chose to bring our sample to temperatures where free flowing liquids were present, which, if done under ground-based conditions, resulted in gross-phase separation and the inability to recover significant superconducting phase. Unwanted reactions with container walls were eliminated by the double float zone. The sheet geometry gave up to 18-gram samples processed with each parabola.

The short time at 0g imposed some processing restrictions. Rapid heating and cooling ramps were needed; thus, the samples were plagued by fractures that we believe are the cause for the low critical currents. The short period of zero gravity necessitated a 1-g anneal that did not

Figure 11: Microstructure in 0g processed, unannealed 29 wt % Ag/Y123 composite processed at a heating rate of 40 °C/s showing silver creep at the right edge.

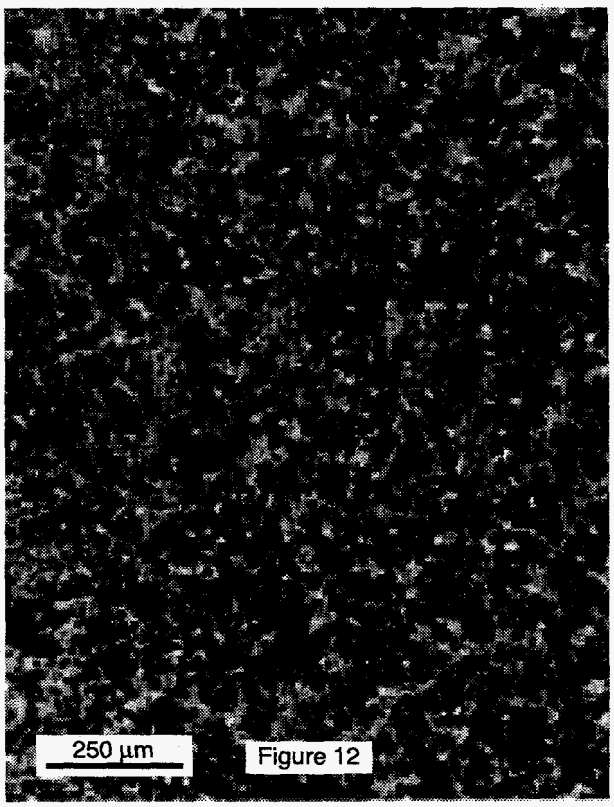

Figure 12: Microstructure in 0g processed, unannealed 29 wt % Ag/Y123 composite processed at a heating rate of 100 °C/s.

erase the microstructural benefit imparted by the 0g processing; thus, long-duration ground-based treatment could follow without invalidating the 0g processing.

Composition uniformity in the unannealed material was sufficient so that a singe 1-g anneal could recover a nearly 100-volume percent superconductor. The ability to achieve 0g melt texturing in the SFZ furnace was demonstrated with a factor of three enlargement in grain size over the 1-g controls but low critical currents resulted from thermal stress-related cracking. In Ag/Y123 composites, dispersion of the small silver grains and the sharpness of the superconducting transition were enhanced.

A number of points unique to a zero-gravity environment that overcame some of the limitations imposed by 1-g processing were demonstrated. Problems with phase inhomogeneities and unwanted reactions with container walls were reduced below our detection limit. Probems with waiting months to years for only a few zero-gravity processed samples were eliminated by building the Sheet Float Zone furnace that flew on the NASA KC-135 and produced hundreds of samples within a period of a few weeks.

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Figure 13: Microstructure in 1-g processed, unannealed 29 wt % Ag/Y123 composite processed at a heating rate of 100 °C/s.

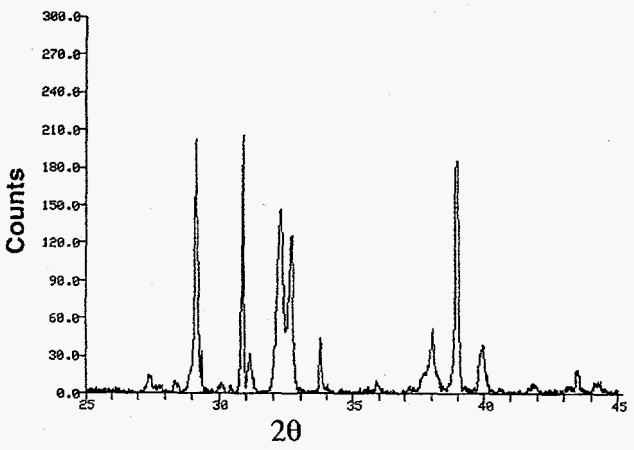

Figure 14: X-ray diffraction spectrum given for unannealed, 0g-processed Y123 on single crystal silicon.

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