The purpose of this Communications section is to provide accelerated publication of important new results in the fields regularly covered by Journal of Materials Research. Communications cannot exceed four printed pages in length, including space allowed for title, figures, tables, references, and an abstract limited to about 100 words.

Preferred orientation of BSCCO via centrifugal slip casting

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Due to the highly anisotropic properties of BSCCO superconductors, the bulk properties of these materials can be greatly affected by preferential orientation. Substantial c-axis orientation normal to the desired direction of current flow has been demonstrated by centrifugally slip casting lead-doped BSCCO-2223. The strong preferred orientation developed in the centrifugally slip-cast material demonstrates high critical current potential.

Limitations to attaining high critical current density (J_c) in bulk polycrystalline high-T_c superconductors include crystallographic J_c anisotropy,[1] superconducting weak links at grain boundaries,[2] and flux line depinning above the irreversibility line.[3] Many of these materials exhibit anisometric grains which can result in low bulk density, further limiting J_c. Texturing anisometric high-T_c superconductors provides a means for crystallographic alignment, suppressing weak link effects, and increasing bulk density, thereby improving superconducting properties.

Many researchers have focused on manufacturing techniques geared toward developing preferred orientation in superconducting materials.[4] Most of these methods are complex and employ liquid phase or powder processes that have limited potential for introducing preferred orientation in bulk components.[5,6] Melt quenching typically produces a thin amorphous layer which must then be annealed to induce crystallization and grain orientation.[5,7] The popular powder-in-tube process involves multiple rolling and annealing steps to produce textured wires or tapes.[8] Rolling and other deformation processes induce high stresses, and may rely on the fracturing of particles to induce texture.

Traditional powder routes for producing oriented microstructures in materials containing anisometric grains include cold pressing,[9] tape casting,[7] and conventional slip casting.[1] Cold pressing of high aspect ratio powders induces texture by forcing the particles to deform, fracture, and/or rotate so that the long direction of the particles is perpendicular to the pressing direction. In tape casting, shearing action aligns the suspended anisometric particles within the slip.[7] Conventional slip casting offers two mechanisms for producing texture. Plate-like particles tend to settle from suspension with their long dimensions perpendicular to the settling direction[1] and capillary fluid flow around settled particles further aids in alignment. Slip casting can produce complex shapes and may not necessitate multiple heat treatments or subsequent deformation processing.

Centrifugal slip casting of anisometric superconductors enables texturing of tube-shaped components which could be internally cooled or serve as magnetic shields. Centrifugal slip casting utilizes the orientation methods involved in conventional slip casting, but also involves additional g-loading to produce higher green densities and strengths. This study investigates the particle alignment in $(Bi,Pb)_2Sr_2Ca_2Cu_3O_x$ (BSCCO-2223) tubes produced by centrifugal slip casting and the effect of alignment on the superconducting properties.

Nitrate-derived Pb-doped BSCCO precursor powder was calcined in an alumina boat for 294 h in air at 860 °C. This powder was then ground using an agate mortar and pestle and stereologically measured to have an average particle intercept size of $\approx 10\ \mu m$. The powder was analyzed using SEM and x-ray diffraction and was found to consist mainly of 2223 platelets, with small amounts of secondary phases. The powder was suspended at a solids fraction of 0.33 in anhydrous hexane[10] using ≈ 4 wt.% of a commercial dispersant (KD3, ICI Specialty Chemicals, Los Angeles, CA). The slurry was thoroughly ultrasonicated (Microson™ XL-2000, Heat Systems, Inc., Farmingdale, NY), yielding a slip having a viscosity similar to vegetable oil.

Centrifugal slip casting was performed on a variable speed wood lathe. The lathe was used to rotate a plaster mold encased in an aluminum sleeve having Teflon end-caps as shown in Fig. 1. The mold was made by mixing 10 parts plaster of Paris with 7 parts de-ionized water (by weight), casting this mixture around a 10 mm

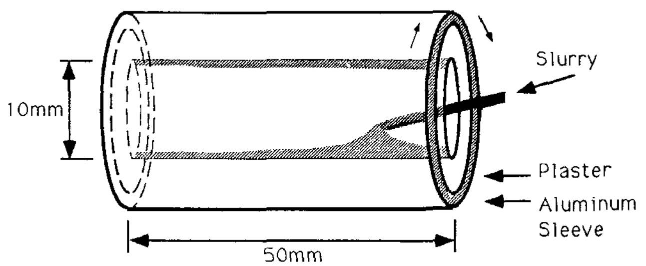

FIG. 1. Schematic diagram of centrifugal slip casting mold assembly.

diameter axial mandrel in the aluminum sleeve, and allowing it to set. The plaster mold was then removed from the aluminum sleeve and dried in air.

In order to facilitate release of the casting from the mold surface, the inside surface of the mold was lined with ashless filter paper (Whatman 42, Whatman International, Maidstone, England). The mold was rotated at 1725 rpm, providing a force of 16.6 *g*'s at the mold wall. The BSCCO-containing slurry was introduced into the mold in three separate injections using a syringe in order to supply enough slip without spillage. The mold was rotated until the entire slurry was cast against the mold wall. The casting was then removed from the mold, and the filter paper was stripped from the BSCCO greenbody. Some damage occurred during this process, resulting in fracture of the tube. Several pieces of the BSCCO greenbody were fired on alumina in air at 840 °C for 3 h. Upon sintering, an average 10% linear *shrinkage* in the thickness was measured, suggesting a beneficial effect of greenbody orientation.

Axial and radial sections of the sintered BSCCO casting were mounted in an acrylic mounting compound (811-202, Leco, St. Joseph, MI) and polished through to 0.05 μm alumina. Figure 2 shows optical micrographs of an axial section of the BSCCO casting and a radial section of a sample of the same powder that was cold-pressed under 50 MPa for 5 min and identically fired. The bulk densities of the centrifugally slip-cast and cold-pressed samples were stereologically measured to be approximately 71% and 58% of theoretical, respectively. The higher density in the centrifugally cast sample is attributed to increased grain alignment. An average grain length of approximately 10 μm was measured from the micrographs, indicating minimal grain growth during sintering. Relatively low grain boundary area is present in either specimen, as indicated by nearly complete penetration of the mounting compound.

As shown in Fig. 2, the centrifugally slip-cast sample appears highly oriented. X-ray analysis of the centrifugally slip-cast and cold-pressed specimens is not a very useful comparison due to the curved geometry of the centrifugally cast sample. In addition, x-ray measurements of texture in these materials are inherently difficult due to peak overlapping and uncertain structure

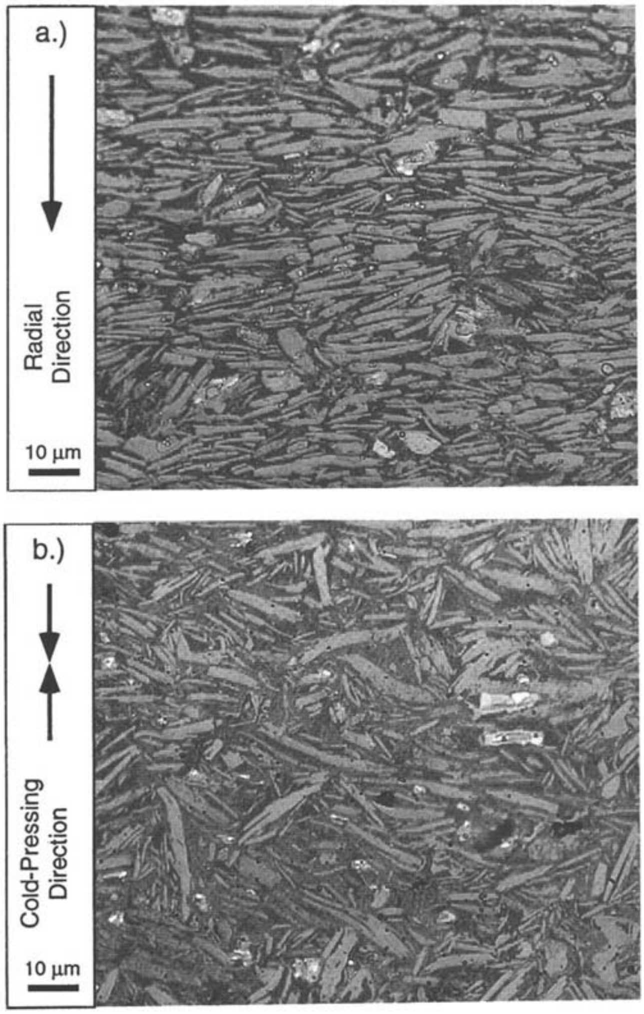

FIG. 2. Typical cross section of sintered BSCCO-2223 platelets aligned via (a) centrifugal slip casting and (b) cold-pressing.

factors. The {0 0 10} and {0 0 12} peaks ($2\theta \approx 24°$ and 29° for Cu K_{α}, respectively) are relatively lower for the highly oriented centrifugally slip-cast sample than for the more randomly oriented cold-pressed sample, as shown in Fig. 3. This result suggests that use of x-ray diffraction for texture quantification is inconclusive for this comparison.

Due to difficulties in x-ray texture quantification of tubes, a modified quantitative stereological technique[11] was applied. Stereological texture analysis of the two samples indicates that the centrifugally slip cast sample is five times more oriented than the nearly randomly oriented cold-pressed sample. The data shown in Fig. 4 describe the distribution of grain tilt angles in the casting from tube tangency or from the compaction plane in the cold-pressed sample. Two extensions beyond the previous analysis technique[11] are employed. First, random grid placement avoids preferential selection of grains for measurement. Second, the average tilt of grains is measured by recording the endpoints of each grain in order to accommodate grain bending.

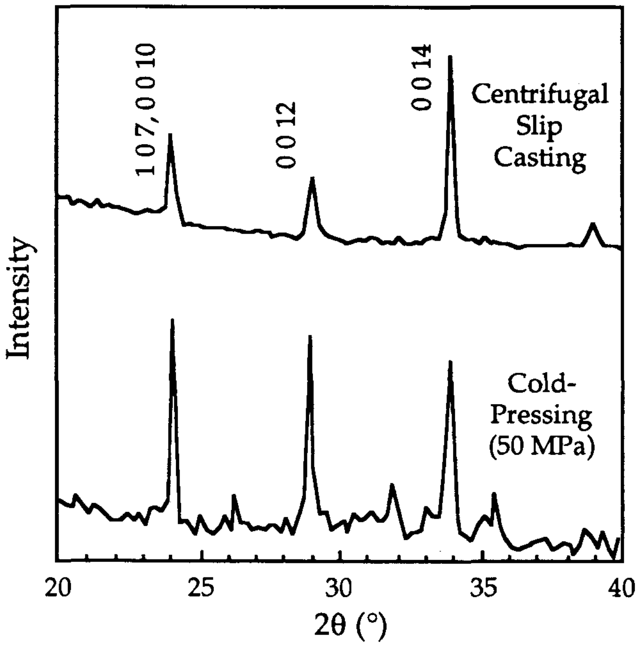

FIG. 3. X-ray diffraction patterns of centrifugally cast and cold-pressed BSCCO-2223 samples.

Magnetization measurements were made on a Superconducting QUantum Interference Device (SQUID) magnetometer (Quantum Design, San Diego, CA). A superconducting transition onset temperature $T_c \approx 110$ K was determined from low field magnetization measurements as a function of temperature. Using the Bean critical state model,[12] the field dependence of the critical current density $J_c(H)$ at $T = 5$ K was determined for the centrifugally slip-cast sample from

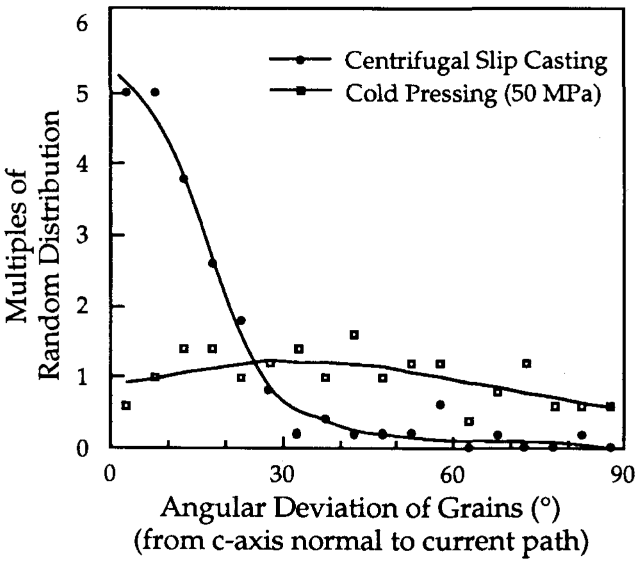

FIG. 4. Orientation histogram developed through stereology comparing typical cross sections of centrifugally slip cast and cold-pressed samples.

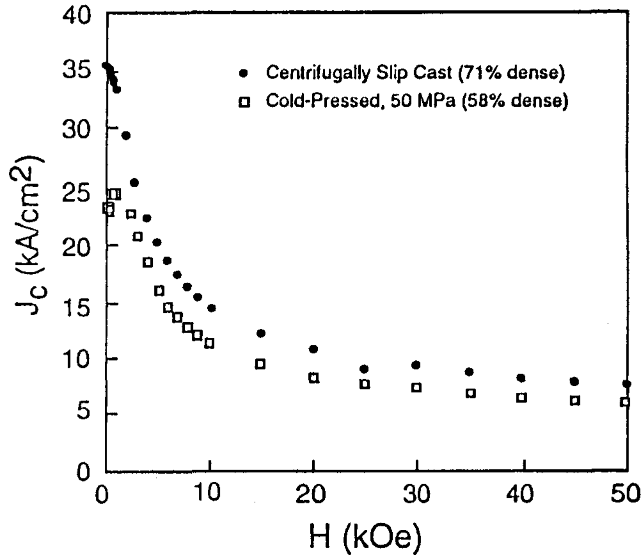

FIG. 5. Plot of $J_c(H)$ at $T = 5$ K for centrifugally cast and cold-pressed samples.

$M(H)$ measurements. The macroscopic dimension of the square sample that was perpendicular to the applied field (2.5 mm) was used to determine the $J_c(H)$ shown in Fig. 5. A less conservative J_c determined on the basis of grain size would be greater by over two orders of magnitude. Despite the damage introduced during handling of the greenbody tube, Fig. 5 shows that after sintering, $J_c(H)$ is approximately 30% higher than for the cold-pressed sample.

After crushing the samples into powder, $M(H)$ was remeasured and found to be lower by a factor of two over the full field range for each sample. The field dependence of J_c before and after crushing was essentially identical after adjusting for the factor of two, suggesting that the field dependence is a property of individual grains. If grains had been well coupled in the bulk samples, the signature of the decoupling should have been evident in the field dependence of J_c

The concept of colloidal texturing based on the hydrodynamic alignment of platelet-shaped particles was demonstrated for BSCCO-2223. Due to the curvature of the sample and the involved x-ray powder diffraction pattern of BSCCO-2223, stereology was used as an alternative method of texture quantification. Stereological analysis shows that the BSCCO particles aligned via centrifugal slip casting are five times more oriented with c-axis orthogonal to the axis of rotation than are the platelets aligned via cold-pressing. Due to the anisotropy of BSCCO, this increased alignment in the slip casting has a significant effect on the densification that takes place during sintering.

The alignment of BSCCO-2223 grains by centrifugal casting may provide a means for producing highly oriented bulk superconducting components without melt-based processing stages. Since J_c is linearly dependent on bulk density, the 22% higher density in the centrifugally slip-cast sample compared to the cold-pressed sample accounts for a significant portion of the overall improvement. The remaining improvement is due to substantial c-axis preferred orientation.

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