TEXTURED CERAMIC TUBES VIA CENTRIFUGAL SLIP CASTING

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ABSTRACT

Unique properties can be developed in materials using centrifugal processing. Highly anisotropic, laminated, gradient and oriented materials can be readily produced using the added gravitational acceleration to control particle arrangement in slurry processing. In addition to the production of oriented microstructures, objects that have cylindrical symmetry can be produced. In this paper, the process of centrifugal slip casting is discussed, two examples of centrifugal slip cast parts are shown for materials in which grain growth is anisotropic, and resulting microstructures are described. The results given in this paper demonstrate that initial green state density can have a strong effect on the development of preferred orientation during grain growth.

Keywords: superconductor, alumina, ceramic, centrifugal slip casting

INTRODUCTION

Centrifugal slip casting (CSC) has been employed to make tube-shaped ceramic materials in production of both monolithic and layered superconducting and structural ceramic materials. Besides the potential for development of preferred orientation in plate- or whisker-like powders, the process also enables the production of gradient or layered microstructures depending on the methods of feeding powder slurries into molds. In this paper, CSC is described as are challenges posed to evaluating preferred orientation in such materials. Texture results from processing of a Bi-based superconductor and alumina/alumina platelet composites are discussed.

BACKGROUND

CSC is a technique for powder processing that combines the effects of slip casting and centrifugation [1-7]. In slip casting, consolidation takes place as fluid is removed through capillary action into a porous mold. Particles within the slip move with the suspending fluid until reaching the mold wall, at which point they become consolidated. In centrifuging, the particles within the slip move through the fluid at a rate dependent upon the G-loading and particle drag [3]. By allowing the particles to move through the liquid, the particles can become oriented before reaching the mold wall or the previously consolidated material. Thereby, the possible

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orientation and fluid-dynamic effects can be tailored to achieve the desired component microstructure. One distinct advantage of the combined action of these two processes, viz. CSC, is that the fluid removal process and the particle orientation can take place simultaneously. Thus, in CSC no supernatant fluid must necessarily be separately drained or siphoned away, which in the processing of thin laminates could damage the prior deposited layers. CSC enables microstructural design of the material in terms of phase ratio, layer thickness, phase gradient and morphological or crystallographic orientation. In order to successfully carry out CSC, mold shape, rotational velocity, casting radius, powder contents, particle density, particle morphology, particle size, particle size distribution, suspension viscosity and interparticle surface forces all play a role [7].

The application of increased G-loading in CSC is based upon centripetal acceleration, the acceleration inward on a body constrained to moving a circular path. The radial acceleration, α, is controlled by both the radius, r, and rotational velocity, ω, of the rotating body through

$$\alpha = \omega^2 r$$

where the relationship between G-loading, G, is given as

$$G = \alpha/g$$

with g = gravitational acceleration, 9.81 m/sec². In centrifugal slip casting the driving force for consolidation of particles is highest at the mold wall or largest radius and decreases linearly toward the center of rotation.

CSC produces a preferred orientation during greenbody formation by anisometric powder particle motion through the liquid and by rearrangement at contact with the mold wall or prior deposited materials. Interference drag can occur, allowing control over powder particle segregation. The Reynolds number, Re, for a particular flow situation allows the prediction of particle rotation within the fluid. Re is a non-dimensional parameter used to describe fluid flow in terms of fluid density, ρ, flow velocity, υ, a characteristic length of the body, l, and the kinematic viscosity, v, of the fluid as

$$Re = \rho \upsilon l / v$$

In the context of this discussion, the characteristic length, l, is the long dimension of the particle and υ is the terminal velocity of the particle in the fluid. Within this context, the nature of platelet motion can be described based upon experimental observations [8-11]. For Re<1, flow is steady and a platelet does no orient relative to flow, and thus should maintain its initial orientation as it settles, until contact with the mold wall changes the orientation. At 1<Re<100, suspended platelets will orient perpendicular to the direction of flow, producing a textured greenbody upon casting. However, at Re>100, vortex shedding produces platelet wobbling or pitching motions and can result in platelet spinning or tumbling at Re>500.

PROCESSING

The materials and slurries employed for both types of materials discussed in this paper are discussed in greater detail in related papers [1-3,7]. For the superconducting materials, suspensions with viscosities equal or lesser than that of vegetable oil ($\approx$0.1 Pa•sec) were

sequentially injected into the mold cavity shown in Figure 1. A layer of slip containing silver powder was injected first, followed by injections of a Bi-based superconductor (BSCCO) slurry and then followed with an injection of another layer of silver slurry. The silver layers are employed to enhance mechanical integrity of the intermediate and final part. Each layer was allowed to cast completely before introduction of additional slip. The mold, mounted on a lathe, was rotated continuously until all layers were cast. The samples yielded tubes with approximate dimensions of outer diameter≈15mm, length≈38mm, silver cladding thickness≈100μm and superconductor thickness≈1.5mm. The mold and casting were then removed from the stainless steel sleeve. The Ag-cladding layers were partially sintered to prevent crack initiation upon mold removal. After removal from the mold some samples were mechanically consolidated via cold-isostatic pressing (CIP). CIP was conducted using evacuated, double layers of latex on each tube. Final sintering was conducted on all specimens at 825°C in flowing 5% oxygen in argon. Figure 2 shows a photograph of a silver-clad tube following production.

Suspensions containing solid volume fractions of 10% Al_2O_3 platelets, with the balance of the solid fraction Al_2O_3 powder, were injected into rotating plaster of Paris molds similar to those used for BSCCO. Parts were sintered in air at 1500°C for 2hr after the specimens were dried and removed from the mold. Figure 3 shows a photograph of an alumina tube following production and the accompanying cross-section microstructure of the tube from an SEM micrograph.

MICROSTRUCTURE

The microstructures of all BSCCO tubes and the alumina/alumina platelet tubes had distinctive preferred orientations similar to those shown in Figure 3. From such micrographs, it is often possible to provide an estimate of the degree of orientation of the platey grains relative to the tube surface tangent by evaluating tube cross-sections. Results of such a quantitative evaluation for the BSCCO material are shown in Figure 4(a) and (b). The orientation distribution shown here was normalized by placing data into five degree bins, to which were assigned a value corresponding to the grain fraction within that bin relative to the total number of grains measured. If all orientations were within a single bin, the maximum value of MRD given would be 18.

From this semi-quantitative approach, it is possible to glean differences in the degree of texturing from different processing histories. It is clear from comparison of parts (a) and (b) of Figure 4 that the samples that had nearly double the densities from CIP at 50MPa did not become as oriented as the less dense samples during high temperature anneals. Since the textures of samples annealed for long times are derived primarily from grain growth, it is not surprising that grain growth was also nearly insignificant in the CIP samples. Additionally, very little densification is apparent following long term anneals for both types of BSCCO tubes.

For the alumina/alumina platelet materials it is clear that the small fraction of platelets were initially strongly oriented and then grew, consuming the alumina powder matrix. Unfortunately, for this particular composite, sintering temperature, and sintering time, densification did not occur quickly enough before the platelet grains grew, inhibiting pore elimination. That little densification occurs during sintering of the BSCCO materials is therefore not surprising, since the initial material consists entirely of plate-like grains that have strong growth anisotropy. Thus, the optimization of microstructure wherein a dense and also oriented material is desired can be suppressed by strong grain growth anisotropy. Production of dense, oriented materials with strong grain growth anisotropy may require a composite approach to processing wherein a mixture of platelet grains and finer, more equiaxed grains could be employed along with isostatic pressing to obtain high densities and platelet grains with strong orientations in the green state. Subsequent grain growth that resulted in microstructures with even as great a porosity as that

shown in Figure 3 would be a significant improvement over those produced by most BSCCO processing routes.

ACKNOWLEDGMENTS

REFERENCES

1. G. A. Steinlage, R. K. Roeder, K. J. Bowman and K. P. Trumble, J. Mater. Res., **9** [4] 833-836 (1994).
2. G. A. Steinlage, K. J. Bowman and K. P. Trumble, U. S. Patent Pending.
3. R. K. Roeder, G. A. Steinlage, K. J. Bowman and K. P. Trumble, J. Am. Ceram. Soc., **78** [9] 2367-73 (1995).
4. A. Royer, J. Mater. Shaping Tech., 197-209 (1988).
5. W. A. Daniel, U.S. Patent No. 2,962,790 (1960).
6. W. A. Daniel, U. S. Patent No. 3,041,699 (1962).
7. G. A. Steinlage, et al., submitted to Ceramic Bulletin.
8. W. W. Willmarth, et al., Phys. of Fluids, **7** [2] 197-208 (1964).
9. E. S. Pettyjohn and E. B. Christianson, Chem. Eng. Prog., **44** [2] 157-72 (1948).
10. V. J. Schmeidel, Physik. Zeit., **29** 593-610 (1928).
11. L. Squires and W. Squires, , Trans. Am. Inst. Chem. Engrs. **33** [1] 1-12 (1937).
12. A. Acrivos, et al., J. Fluid Mech., **34** 25-48 (1968).
13. F. T. Smith, J. Fluid Mech., **155** 175-191 (1985).
14. J. D. Hudson and S. C. R. Dennis, J. Fluid Mech., **160** 369-83 (1985).
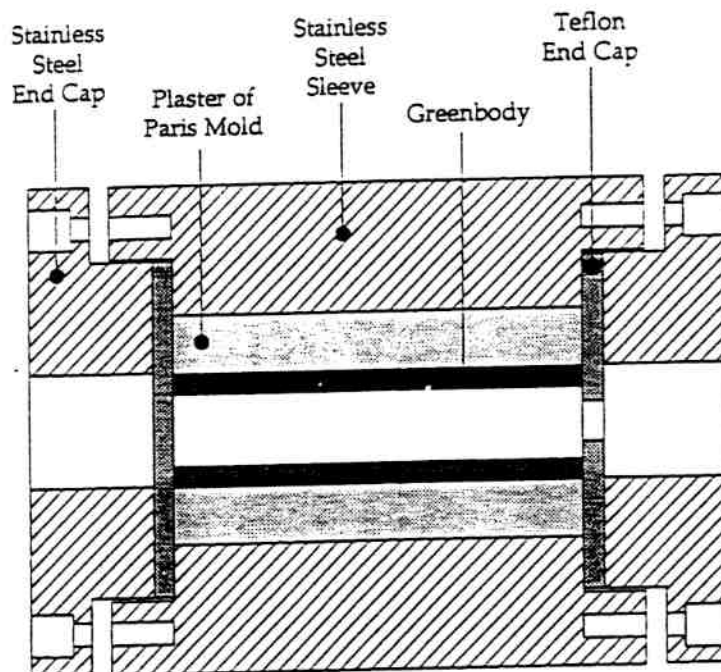15. D. B. Ingham, T. Tang, and B. R. Morton, J. Fluid Mech., **210** 281-302 (1989).

Figure 1. Schematic of the centrifugal slip casting mold assembly.

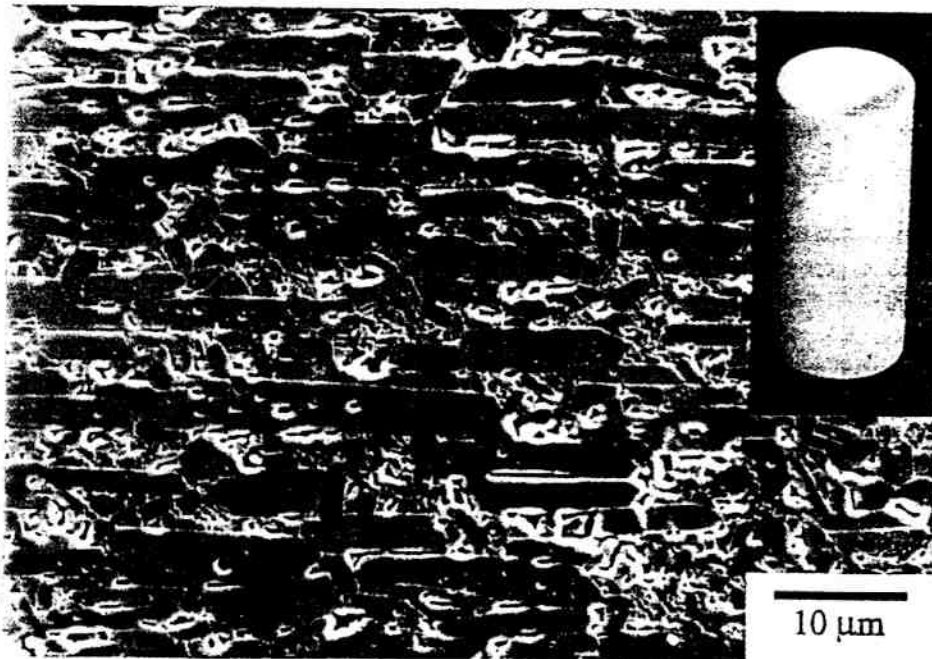Figure 2 Photograph of sintered centrifugally slip cast Ag-clad BSCCO tube (major divisions in centimeters.

Figure 3 Microstructure of a centrifugally slip cast tube, with homogeneousiy distributed Al_2O_3-platelets in an Al_2O_3 matrix.
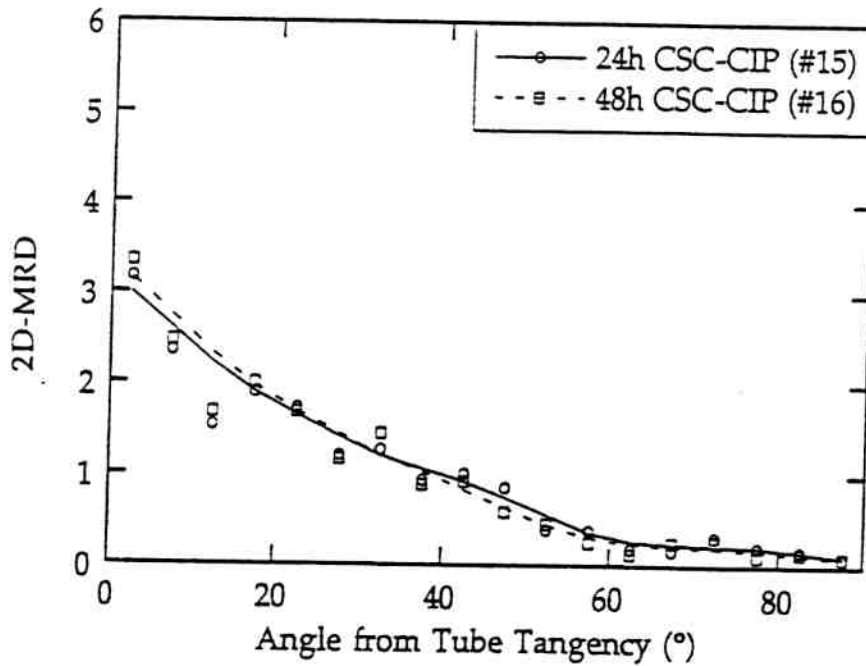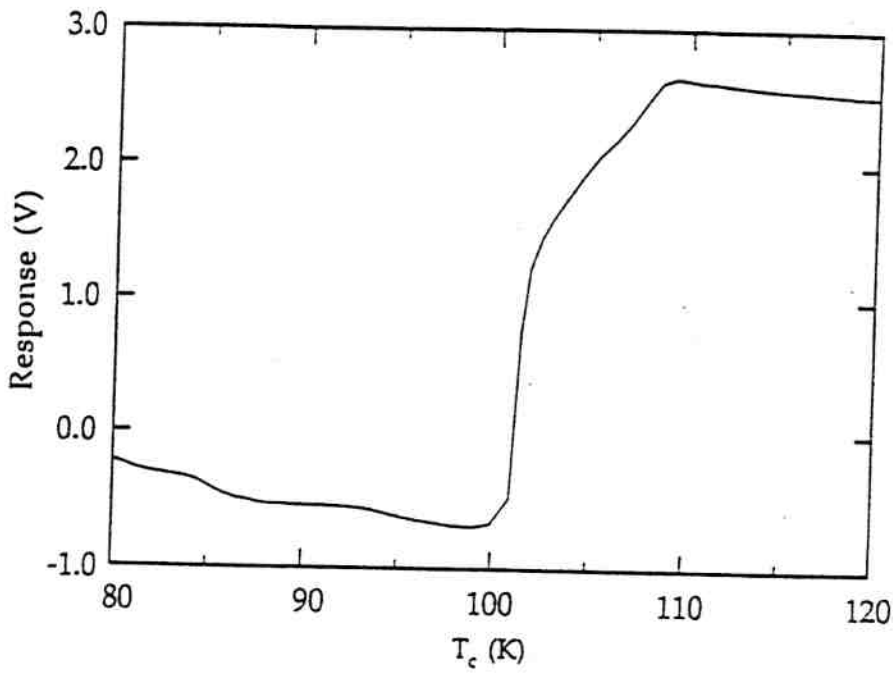
Figure 4 Distribution plot of projected angle from tube tangency for BSCCO grains of (a) centrifugally slip cast Ag-clad BSCOO tubes sintered for 24 and 48 hours and (b) for tubes that were additionally isostatically pressed and then sintered for 24 and 48 hours.

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