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Processing and Properties of Bulk High-Temperature Superconductors

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Processing methods for production of bulk high-temperature superconductors and the superconducting properties that can be achieved are reviewed. Conductors made by conventional ceramic forming and sintering methods can generally sustain only small current densities. Partial-melting and powder-in-tube methods yield superior conductors, but further improvements are necessary before these superconductors can be employed in most large-scale applications.

I. Introduction

The recently discovered high-temperature superconductors have transition temperatures greater than 90 K and can carry large amounts of current in large magnetic fields. Potential large-scale use for these materiais require that the materials be fabricated into shapes such as wires, tapes, or other bulk forms. Fabrication involves synthesis of powders, shape forming, and heat treatment. Challenges are posed to successful fabrication, however, by the brittle natures of the high-temperature superconductors and by the difficulties associated with grain-boundary weak links and flux motion[1].

Several methods are now used to fabricate bulk superconductors[1-3]. The resultant properties are dependent on both methods and materials. A survey is given of fabrication of superconductors based on Y-Ba-Cu-O, Bi-Sr-Ca-Cu-O, and Tl-Ba-Ca-Cu-O. Critical current density (J_c) values are greater than 10^5 A/cm^2 for each material under some conditions of temperature and applied magnetic field. Because the methods and results vary among the three compounds, each will be discussed individually.

II. Processing and Properties of $YBa_2Cu_3O_x$

Fabrication of bulk $YBa_2Cu_3O_x$ (YBCO) conductors generally begins with synthesis of powder. Most powders are prodiiced by reaction between Y_2O_3, $BaCO_3$ or BaO_2 and CuO or Cu_2O [4]. The starting powders are rnechanically mixed until homogeneous. The mixed powders are then heated in an oxidizing atmosphere to a temperature greater than about 800°C. Depending on the temperature, a reaction to form YBCO takes place in one hour to about one day. Low oxygen partial pressures and flowing gas maintained at low total pressure promote rapid reaction to form the superconducting phase. Benefits are derived from the rapid removal of deleterious evolved species[4] and enhanced diffusional kinetics at reduced oxygen pressure[5,6]. The YBCO phase is stable over a relatively narrow range of O_2 pressure, however, and the useful atmospheres generally range from 2×10^2 to 10^5 Pa of O_2 [5,6].

Chemical solution methods, such as coprecipitation, freeze-drying, sol-gel processing, or liquid-mix techniques are also being used to synthesize YBCO[8,9]. In principle, each of these methods is capable of yielding a homogeneous mixture of the constituents on a microscopic scale. Heat treatments similar to those used with mixed-oxide powders are required to produce superconducting YBCO. Advantages of solution-derived powders include introduction of little or no contamination during mixing and the possibility of producing very fine powders. To date, generally because of difficulties in completely removing the solution constituents during heat treatment, solution-derived powders have not produced superconductors with properties superior to those of solid-state-synthesized powders.

Once YBCO powder has been synthesized, it must be consolidated into bulk forms. Techniques such as plastic extrusion and tape casting have been discussed

previously[1,8,9]. In these plastic forming methods, YBCO powders are mixed with appropriate amounts of a dispersant, a binder, a plasticizer, and a solvent that is compatible with the other constituents of the mixture. The resultant mass is formed into the desired shape and heat treated[8,11]. Modest texturing of the YBCO grains generally occurs during forrning, and slight improvements in J_c as compared with, for example, pressed pellets have been reported[S]. In the absence of some type of partial-melt heat treatment, these types of wires and tapes exhibit J_c values less than about 2×10^3 A/cm^2 at 77 K and, owing to weak links at grain boundaries, the J_c drops substantially in an applied magnetic field[8,12]. Because of the low J_c values in field, such conventionally sintered forrns are suitable only for low-current-density applications such as current leads[13].

Significant improvements in J_c at 77 K have been obtained by several groups by packing YBCO powder into a metallic tube and mechanically working the tube by swaging, drawing, extrusion, rolling, or pressing to produce a conductor[1,8]. YBCO reacts with all metals, but it is nearly inert to Ag and Au [14]; therefore Ag tubes are generally used in these techniques. Achievement of high core densities in the unfired state, which allows for the use of low sintering temperatures, has yielded wires with J_c values of R 10^4 A/cm^2 at 77 K in zero field[1]. Ag-clad YBCO wires generally exhibit only slight texturing of grains and, as with other sintered forms of YBCO, the J_c values decrease relatively rapidly in applied magnetic fields[15].

Grain-boundary weak links remain the most severe obstacle to the development of useful bulk YBCO superconductors. Partial-melt heat treatments are capable of eliminating grain-boundary weak links in YBCO[1]. These treatments have been used with and without temperature gradients in the furnace. Monoliths[16-23] or thick films on ceramic substrates[24] can be successfully melt-processed. Thick films can also be processed on metallic substrates, but owing to diffusion of Cu from the YBCO into the metal, heat treatment can be difficult to optimize (Figure 1). In partial-melt processing, a bulk YBCO piece is heated above the peritectic melting point ($\approx 1020°$C in O$_2$) and is cooled either very slowly in a thermal gradient[16,20], or a molten zone is passed slowly along it[21]. Very large grains grow. Because the incongruent melting point of YBCO can be lowered by reducing the oxygen partial pressure[3], many substrates can be used. In addition, off-stoichiometric compositions of YBCO that form a eutectic liquid with Ag can be used to lower the temperature for melt-texturing to $\approx 930°$C [25].

· In the absence of a thermal gradient, large domains of elongated, well-aligned YBCO grains will form upon solidification[1,26]. The transport J_c within each domain may be high, but because of nonsuperconducting phases between the domains, the J_c between the domains is

generally no more than R 10^3 A/cm^2 at 77 K. For well-made melt-textured YBCO, J_c values within the domains, both transport and magnetic[20], have exceeded 4×10^4 A/cm^2 at 77 K in fields greater than 1 T. The high J_c is due to the existence of large, elongated grains separated by low-angle grain boundaries (Figure 2) that are capable of transporting large amounts of currents. In addition, the grains thernselves have high J_c values[1,27]. The high intragranular J_c appears to be related to high defect concentrations within the grains. Flux pinning can come from many defects. Oxygen vacancy ordering appears to improve flux pinning[28]. In addition, melt-texturing produces large concentrations of dislocations and stacking faults, both of which have been shown to increase pinning[20,27]. Precipitates have been shown to increase pinning also. Among those reported to be effective in YBCO are the: Y_2BaCuO_5 phase[18,21]; CuO, either by direct addition[29] or from decomposition of $YBa_2Cu_4O_x$ [30]; SnO_2 [31]; and $BaTiO_3$ [32]

It is noted that the transport J_c values of YBCO with specific second-phase additions have not exceeded that of high-quality melt-textured YBCO without deliberate additions. Transport J_c for a length of wire would appear to be controlled more by grain boundaries than by the intrinsic J_c of the grains. Furthermore, it has yet to be demonstrated that long, continuous lengths can be made by melt-texturing YBCO. Texturing thick films for small-scale applications remains an attractive possibility and use of substrates with higher melting temperatures than that of Ag may be helpful. In addition, use of larger temperature gradients may aid in making longer lengths of wire by drastically reducing the processing times[33].

III. Processing and Properties of Bi-Sr-Ca-Cu · O

The Bi-Sr-Ca-Cu-O (BSCCO) system contains superconductors that have T_c values of about 90 and 110 K. The composition of the 90 K phase is approximately $Bi_2Sr_2CaCu_2O_x$ (2212), although the compound is actually a solid-solution with a fairly large range of cation content. The $Bi_2Sr_2Ca_2Cu_3O_x$ phase (2223), which is stabilized by partial substitution of Bi by Pb and also has a range of solid solution, has been found to consistently have T_c values greater than 100 K [1].

BSCCO powders are commonly synthesized by routes similar to those used for YBCO[1,7,34]. The types and amounts of the many possible phases that exist in the BSCCO system are dependent upon the starting stoichiometry, the processing atmosphere, and the thermal treatment. It has proved to be very difficult to obtain phase-pure powders. In addition, because of very rapid anisotropic grain growth, it has proved to
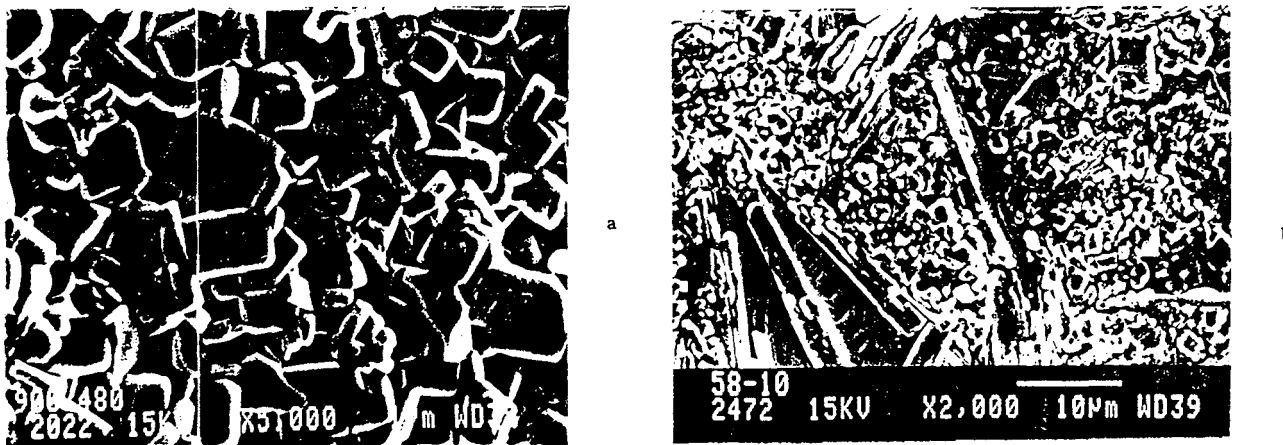
Figure 1: YBCO thick films made by spray pyrolysis: (a) well-made film heat treated in 1% O_2 at 900°C, consisting of textured grains and (b) a film that was heat treated in 1% O_2 at 930°C, which contains extensive Y_2BaCu_5 needles along with the superconducting phase.

be very difficult to sinter BSCCO to high density[35]. Thus, largely because of poor connectivity between most grains, conventionally sintered BSCCO superconductors generally exhibit even lower J_c values than does sintered YBCO.

Novel approaches such as spin coating have been used to produce dense, textured films of BSCCO from solutions. In one example of this approach[36], solutions with stoichiometric compositions Bi:Sr:Ca:Cu = 2:2:1:2 and (Bi, Pb):Sr:Ca:Cu = (1.6,0.4):2:2:3 were prepared by dissolving Bi, Ca, and Cu acetates, Sr (and Pb, in the case of the doped solution) nitrate in glacial acetic acid and water. Changes in pH were achieved with ammonium hydroxide. Various amounts of polyvinyl alcohol were added to change the viscosity of the solutions. Attempts to change viscosity were also made with polyacrylic and tartaric acids, but these acids did not produce homogeneous solutions, and consequently good films were not obtained.

The solutions used to prepare films by spin-coating had a viscosity of $\approx$ 1.00 cP at 30°C. The spinning velocity was $\approx$ 3500 revolutions/min. Ag plates were coated with 10 layers to achieve a thickness after heat treatment of 2-5 μm for both compositions. The films were heated to 600°C after deposition of each layer of coating. Thus, the spin-coating and heating processes were repeated 10 times. The final sintering was conducted at 850°C for periods less than 1 h. The heat treatment atmospheres were air, O_2, or 1% O_2/99% N_2. In all cases, the heating and cooling rates were 0.5°C/min and 2°C/min, respectively.

Figure 3, a scanning electron micrograph of a Pb-doped film made by spin-coating and sintering at 825°C in O_2, shows that long grains ($\approx$ 300μm) originate from a few nuclei and that between the long grains are equiaxed fine grains that are very dense. The best J_c

values obtained, $\approx 10^3$ A/cm^2 at 77 K, were associated with this type of textured microstructure.
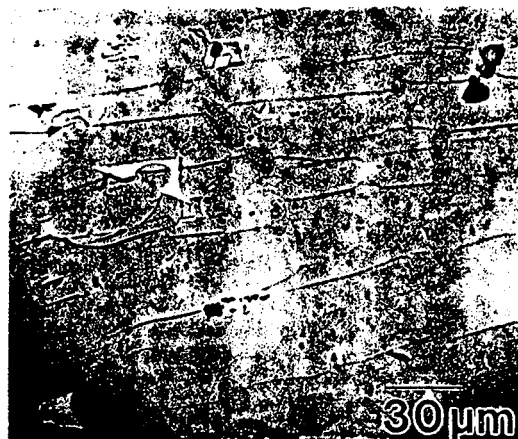

Figure 2: Scanning electron microscopy photograph of long, aligned grains in melt-textured YBCO.

In general, the processing methods for BSCCO superconductors made from synthesized powders are similar to those for YBCO. Because the BSCCO compounds melt at lower temperatures than YBCO, and because of an absence of low-melting-point eutectics in the BSCCO system, melt-processing of BSCCO has proved to be highly effective. The 2212 phase can easily be partially melted in Ag tubes[37-40] or on Ag substrate[41], and thus long lengths can be formed. Similar to the spin-coating process, partial melting produces elongated 2212 grains aligned with the substrate. Contact area between the 2212 and Ag can be increased by fabricating a wire with an Ag core and an Ag sheath[40]. When Pb-doped 2223 is processed with Ag, usually little melting is allowed to take place because of problems
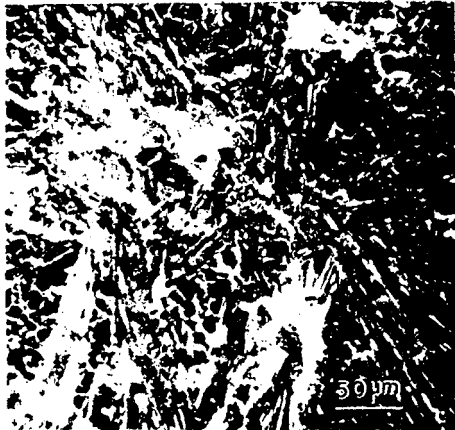
Figure 3: Scanning electron microscopy micrograph of Pb-doped 2223 BSCCO film containing elongated, textured grains.
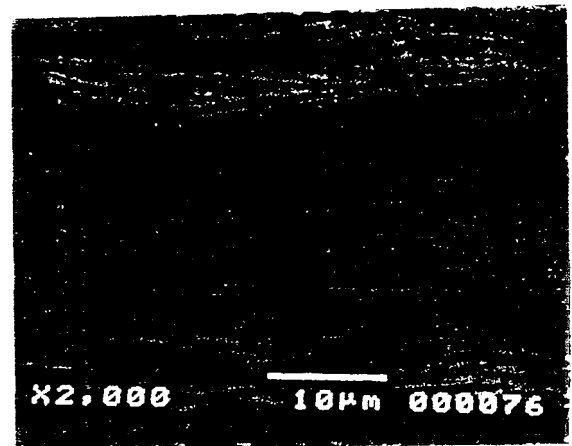

Figure 4: Scanning electron microscopy photograph of aligned grains of 2212 BSCCO formed by flat-rolling in an Ag sheath.

with phase separation[42].

The grain growth inherent in the BSCCO compounds provides an opportunity to mechanically texture tapes and thick films. Combinations of flat-rolling or pressing and heat treatments designed to grow grains result in dense superconductors[40-51] with high degrees of texture (Figure 4). An excellent review of the results produced by powder-in-tube processing of BSCCO superconductors can be found in Ref. 52. In brief, the J_c values at 4.2 K of BSCCO/Ag superconductors can exceed 10^5 A/cm^2 in zero field and 10^4 A/cm^2 in a 20 T field[51]. At 20 K, J_c values can exceed 5×10^4 A/cm^2 in fields greater than 10 T [44]. Flux motion under an applied field is a problem, however, for temperatures greater than $= 30$ K [1,43,44]. At 77 K, J_c values of BSCCO superconductors have reached $\approx 5 \times 10^4$ A/cm^2 in zero field, but because of flux motion, which may be most severe at the grain boundaries, the J_c decreases rapidly in an applied magnetic field[51]. The high transport J_c values at temperatures low enough for flux motion to be minimal appear to be related to the high degrees of texture in BSCCO/Ag superconductors[1]. A simple way of viewing the current transport of BSCCO superconductors is shown in Figure 5. Although current flow may be impeded by high-angle grain boundaries, the current may move along the c-axes of the crystals and thus follow a tortuous path through the BSCCO core[1].

Substantial effort. is now focused on introducing flux-pinning centers into BSCCO superconductors. Some success has been reported. Possible pinning centers include Ca-Cu-O precipitates[53], dislocations[54,55], or radiation-induced defect cascades[56,57]. Elimination of 'flux motion in applied magnetic fields through microstructural control will be required before BSCCO superconductors can be used in most applications at
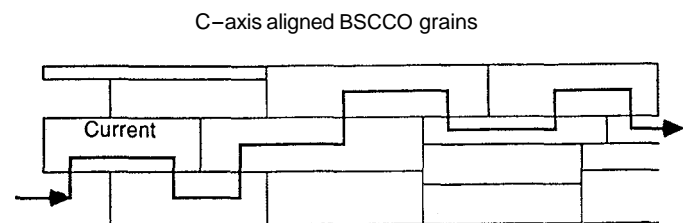
C–axis aligned BSCCO grains

Figure 5: Schematic diagram of current transport in aligned BSCCO superconductors.

temperatures greater than about 20 K [1]. Commercial application of these superconductors may also be hindered by the fact that BSCCO/Ag superconductors have yet to demonstrate good mechanical reliability. For example, it has been found recently that strains of $\approx$ 0.4% cause J_c to reduce by $\approx$ 50% [51]. It has also been observed that Ag-clad BSCCO superconductors are sensitive to thermal cycling.

IV. Processing and Properties of Tl-Ba-Ca · Cu - O

A T_c of 125 K has been reported for the $Tl_2Ba_2Ca_2Cu_3O_x$ phase[58] and there are many superconducting phases with T_c values greater than 100 K in the Tl-Ba-Ca-Cu-O (TBCCO) system[58,59]. Although the basic layered crystal structures of TBCCO are similar to those in the BSCCO system[59], bulk TBCCO has not been textured mechanically to the extents that the BSCCO superconductors have. In addition, bulk processing of TBCCO is entirely different from that of YBCO or BSCCO because of extensive liquid-phase formation. Tl_2O_3 decomposes to Tl_2O above about 850°C

and Tl_2O melts at about 300°C [60]. Thus, upon decomposition, rapid liquid-phase sintering results. Tl also volatilizes at high temperatures. Use of sealed containers, most commonly Au or Pt, improves compositional control by establishing a Tl_2O partial pressure and thus minimizing loss of Tl from the starting mixture. It has also been shown that the compounds with one Tl-O layer suffer less Tl loss than do those with two Tl-O layers [61]. It is possible to use the volatility of Tl to one's advantage by fabricating a wires or tape of the correct Ba-Ca-Cu-O stoichiometry and diffusing vapor-phase Tl inward to form a superconductor [62-64].

Interest in TBCCO superconductors has increased substantially since the recent discovery that the $TlBa_2Ca_2Cu_3O_x$ phase (1223) has an irreversibility line near that of YBCO and much above the lines of the other TBCCO compounds and the BSCCO compounds [65]. The inherent promise of the 1223 TBCCO compound has been demonstrated recently. Thick films of 1223 on polycrystalline Y_2O_3-stabilized ZrO_2 substrates have achieved transport J_c values of greater than $6 \times 1C^4$ A/cm^2 at 77 K [64].

Metal-clad tapes have been made with the $Tl_2Ba_2Ca_2Cu_3O_x$ compound, with Sr substituted for some of the Ba [67,68]. The best J_c at 77 K in zero field was $\approx 10^4$ A/cm^2, but apparently because of flux flow, J_c dropped to $\approx 10^4$ A/cm^2 in a 1 T field. Ag may react more strongly with the TBCCO compounds than it does with either YBCO or the BSCCO compounds [69]. Melting and formation of second phases are promoted. Au is much more inert with respect to TBCCO, and it has been used as a substrate in high-speed zone-melting of TBCCO compounds [68,70]. The microstructures produced to date are similar to those of melt-textured YBCO and the J_c at 77 K has reached $\approx 10^4$ A/cm^2.

It remains to be seen whether the problems associated with Tl volatility and toxicity and the possible incompatibility with Ag can be overcome so that high-quality TBCCO wires or tapes can be fabricated on a commercial basis. The best J_c values to date are superior to those of most YBCO forms and are comparable to those of BSCCO at 77 K.

V. Summary

Each of the high-T_c superconductors presents unique challenges for fabrication of bulk conductors. Recent results of promise include attainment of J_c values of $10^4 - 10^5$ A/cm^2 in highly textured wires or tapes of YBCO, BSCCO, and 1223 TBCCO. Elimination of weak links and production of continuous lengths remain the principal fabrication goals for YBCO. Reduction or elimination of flux motion is the principal goal in fabricating the BSCCO superconductors. The BSCCO compounds texture very well, and weak links do not appear to be a problem at temperatures less than $\approx 20 - 30$

K. Flux motion remains a problem for the TBCCO superconductors also, although the 1223 phase may to a large extent overcome this problem, as demonstrated by recent results [64].

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