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6-4-1993

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Abstract

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Keywords

High temperature superconductors, Critical currents, superconductors

Disciplines

Metallurgy

Comments

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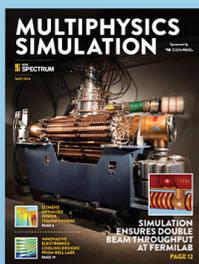
Directional isothermal growth of highly textured $Bi_2Sr_2CaCu_2O_y$

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For $Bi_2Sr_2CaCu_2O_y$ (2212), it is shown that an oxygen gradient, as opposed to a temperature gradient, can be used to produce large bulk forms of the 2212 superconductor with highly textured microstructures from an oxygen-deficient melt held at a constant temperature. Material produced in this manner was found to have transition temperatures between 85 and 92 K, high critical current densities below 20 K, and modest critical current densities at 77 K.

The use of high-temperature superconductors for conductor applications will require material with textured microstructures which eliminate or minimize the high-angle grain boundaries ("weak links") that limit intergranular critical current densities.[1] Several approaches have been used for processing and texturing of the $Bi_2Sr_2CaCu_2O_y$ (2212) superconductor. The coupled use of silver and melt processing in the partially molten state for the production of highly textured wires and tapes has been well documented.[2,3] For larger bulk forms, laser-heated floating-zone or modified Czochralski techniques have been successfully used to directionally solidify 2212 fibers.[4,5] An important aspect of directional-growth techniques from a pure melt is the use of a temperature gradient between the melt and the solidifying material.[6] This method works best for congruently melting compounds. However, the 2212 phase melts incongruently and, as a result, directional growth processes or melt-processing techniques must maintain slow growth or cooling rates, respectively, in order for the peritectic phase to be favored during solidification.[7]

The 2212 phase is not stable in inert atmospheres at high temperatures and decomposes into $Bi_2Sr_{3-x}Ca_xO_y$ and Cu_2O.[8] The solidus is substantially reduced from 895 °C in O_2 to around 750 °C in Ar.[8,9] Solidification studies have shown that the alkaline-earth cuprates, which are products of the peritectic reaction involving 2212, are not present in the absence of oxygen. The resulting microstructure is a divorced eutectic of $Bi_2Sr_{3-x}Ca_xO_y$ and Cu_2O.[8] Discounting the CaO that separates out in the melt, the highest temperature at which crystalline phases exist in Ar is approximately 820 °C. Hence, it should be possible to solidify an oxygen-deficient melt at a constant temperature by simply switching to a high oxygen partial pressure. The oxygen gradient that is set up within the sample is analogous to the temperature gradient used in directional solidification. A related approach was reported for $Bi_2Sr_2Ca_2Cu_3O_y$ (2223) in which samples melted in Ar were oxidized by slowly increasing the oxygen partial pressure from 0% to 100% over a 20–100 h span.[10] However, the slow oxidizing rate used in the latter study does not

permit a large oxygen gradient to be set up in the sample, and the directional growth aspects discussed below were not found. Furthermore, the 2223 phase is not particularly well suited for this process since a very large amount of CaO separation occurs on melting.

Compositions used in this study were initially processed by splat quenching in order to produce a homogeneous starting material that is oxygen deficient.[11] Ground powders of the glassy material were placed in alumina or Ag boats and melted in Ar at 900 °C for 10–30 min. Samples were then cooled to a temperature between 870 and 825 °C. After the temperature had stabilized, the gas flow in the furnace was switched to oxygen and the samples were held at this temperature for 50 or 10 h. Gas flows were set to change the equivalent volume of the furnace tube every 0.5 h. Samples were examined by optical and scanning electron microscopy (SEM). Quantitative chemical analysis of the phases present in these samples was performed in the SEM by energy-dispersive spectroscopy (EDS).[12] Transition temperatures were determined from the extrapolated onset of the diamagnetic signal in a SQUID magnetometer. Critical current densities were determined from hysteresis loop measurements on 3-mm disks cut from the sample and by pulsed dc transport measurements at 77 K on the whole sample.

Large-grained, textured microstructures were produced with the directional isothermal growth (DIG) process as evidenced by the fractured and polished cross sections shown in Figs. 1(a) and 1(b), respectively. The growth direction of the 2212 grains is from the surface downward (left to right in Fig. 1). A surface layer is evident in both micrographs of Fig. 1 and was common to all directionally solidified samples. This layer is caused by the separation of CaO in the melt, which subsequently accumulated near the surface. Phases identified in this layer by compositional analysis were $Bi_2Sr_{4-x}Ca_xO_y$, $Sr_{1-x}Ca_xCuO_y$, CaO, and 2212. The presence of these phases is consistent with expected phase assemblages for a Ca-rich composition.[13,14] Figure 1(b) shows that the nucleation and growth of the large oriented grains seen in the fracture cross section starts at the bottom of the surface
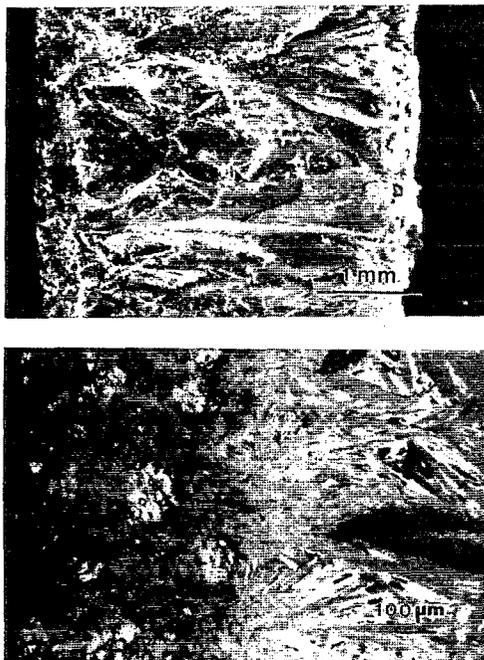
FIG. 1. Secondary and backscattered electron micrographs, respectively, of (a) fractured and (b) polished cross sections of directionally grown material with an overall composition of $Bi_{2.15}Sr_2Ca_{0.85}Cu_2O_y$. The growth direction is from left to right.

layer. As shown in Figs. 1(a) and 1(b), these grains grew with the orientation of their c axes (short dimension of the grains) approximately perpendicular to the growth direction. When viewed along the growth direction, the c axes of these grains are found to be randomly oriented in the plane perpendicular to the growth direction as shown in Fig. 2. The texturing that results during the DIG process is aided in part from the anisotropic crystallographic growth rates of the 2212 phase.

Single-phase 2212 material was not produced in these samples. The backscattered electron micrograph of Fig. 1(b) clearly shows two phases below the surface layer which were identified as the low temperature superconductor $Bi_2Sr_{2-x}Ca_xCuO_y$ (2201) and 2212. CuO was also found in this region. The presence of the 2201 phase is a direct result of CaO separation in the melt. This problem

FIG. 2. Optical micrograph of a directionally grown sample with an overall composition of $Bi_2Sr_{2.15}Ca_{0.85}Cu_2O_y$. The growth direction is into the micrograph.
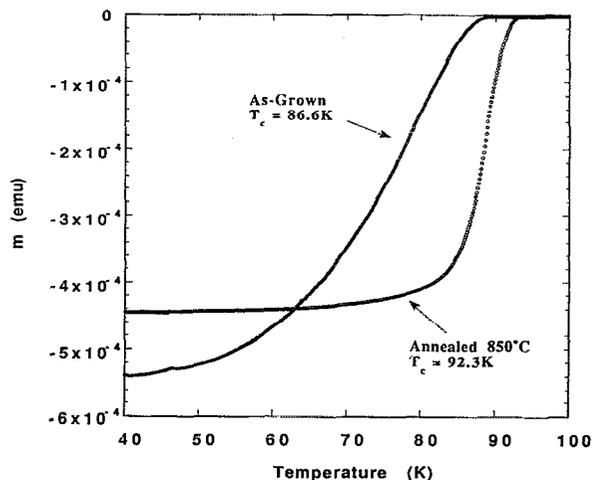

FIG. 3. Magnetization data on as-grown and annealed directionally grown samples with an overall composition of $Bi_2Sr_{2.15}Ca_{0.85}Cu_2O_y$.

was minimized, but not eliminated, by starting with a Sr-rich composition such as $Bi_2Sr_{2.15}Ca_{0.85}Cu_2O_y$. In order to determine the approximate composition of the liquid from which grain growth occurs, a sample of this Sr-rich composition was melted in a small Ag boat in Ar at 900 °C, cooled in Ar to 850 °C, and then quenched on a large metal block. SEM analysis revealed that almost all CaO particles that formed in the melt segregated to the surface region. Chemical analysis in the SEM of the material below this surface layer revealed an overall Ca-poor, Cu-rich composition of $Bi_{2.09}Sr_{2.15}Ca_{0.52}Cu_{2.24}O_y$. Hence, the effective composition from which grain growth occurs lies in a three-phase region of 2212, 2201, and CuO.[14]

Regardless of the starting stoichiometry, the measured compositions of the 2201 and 2212 phases were nearly the same. The average of 105 separate measurements of the 2212 phase from 14 samples of various starting compositions around the 2212 stoichiometry was $Bi_{2.15}Sr_{2.02}Ca_{0.83}Cu_{2.00}O_y$. This composition lies on the Sr-rich end of the 2212 solid solution range.[14] The average of 45 separate measurements of the 2201 phase from these samples was $Bi_{2.18}Sr_{1.56}Ca_{0.24}Cu_{1.02}O_y$. For compositions around the 2212 stoichiometry, the separation of CaO during melting in Ar results in a Ca-deficient liquid composition lying between the 2212 and 2201 solid solution regions. Hence, contact is always made with the Sr-rich end of the 2212 solid solution region during solidification.

Transition temperatures of the as-grown material were found to lie between 85 and 90 K. T_c could be raised to 92 K by a subsequent annealing of the material in oxygen, as shown in Fig. 3. However, no change was observed in the microstructure of the material or measured compositions of the 2212 and 2201 phases even after anneals of 100 h. Critical current densities shown in Fig. 4 were calculated from hysteresis loop measurements at 5, 10, and 20 K on a 3-mm disk (field applied perpendicular to the disk) cut from the annealed sample of Fig. 3. J_c values of the as-grown sample were found to be approximately half of those of the annealed sample. The critical currents were assumed
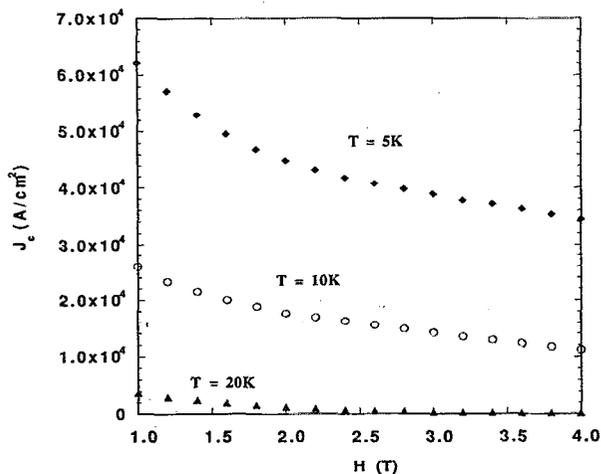
FIG. 4. Critical current densities calculated from hysteresis loop measurements via the Bean model on the annealed sample shown in Fig. 3.

to be intergranular and the entire sample size was used as the critical dimension in the calculation of the current densities from the Bean model.[15] The intergranular nature of the critical currents is supported by the fact that quartering of the disks reduced the M-H loops by a factor of approximately 2. Transport measurements at 77 K in self-field on 2212 bars ($\approx$90 mm$\times$15 mm$\times$4 mm) grown by the DIG process revealed J_c values ranging between 150 and 450 A/cm^2. The large decrease in J_c with increasing temperatures and/or field above approximately 20 K is typical of the 2212 superconductor.

In summary, it is shown that an oxygen gradient can be used to directionally solidify large-grained, textured bulk forms of the 2212 superconductor from an oxygen-deficient melt at a constant temperature. Transition temperatures were found to be in the range of 85–90 K. This material was found to have high intergranular critical current densities below 20 K and modest values at 77 K. The composition of the 2212 phase was always found to be approximately $Bi_{2.15}Sr_{2.02}Ca_{0.83}Cu_{2.00}O_y$. Samples pro-

duced in this manner were not single phase due to CaO separation in the melt. However, improvements in the material and superconducting properties are expected when problems associated with CaO separation in the melt are minimized or eliminated. The relatively simplistic nature of the DIG process can be easily scaled up to produce large rigid conductors.

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