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Superconducting properties of Gd-Ba-Cu-O single grains processed from a new, Ba-rich precursor compound

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Abstract Gd-Ba-Cu-O (GdBCO) single grains have been previously melt-processed successfully in air using a generic Mg-Nd-Ba-Cu-O (Mg-NdBCO) seed crystal. Previous research has revealed that the addition of a small amount of BaO_2 to the precursor powders prior to melt processing can suppress the formation of Gd/Ba solid solution, and lead to a significant improvement in superconducting properties of the single grains. Research into the effects of a higher Ba content on single grain growth, however, has been limited by the relatively small grain size in the earlier studies. This has been addressed by developing Ba-rich precursor compounds Gd-163 and Gd-143, fabricated specifically to enable the presence of greater concentrations of Ba during the melt process. In this study, we propose a new processing route for the fabrication of high performance GdBCO single grain bulk superconductors in air by enriching the precursor powder with these new Ba rich compounds. The influence of the addition of the new compounds on the microstructures and superconducting properties of GdBCO single grains is reported.

1. Introduction

Melt processed (LRE)-Ba-Cu-O single grain superconductors [(LRE)BCO], where LRE is a light rare earth element such as Nd, Sm, Eu and Gd, attract significant research interest because of their higher critical current densities, J_c's, and higher irreversibility fields than Y-Ba-Cu-O (YBCO) [1-3]. The recent development of a generic seed [4] has made processing of (LRE)BCO single grains in air easier, and has generated renewed interest in these materials. To date, GdBCO single grains have been melt processed in an air atmosphere using the generic seed by the so-called cold seeding method. In addition, BaO_2 has been used to suppress Gd/Ba substitution in the Gd solid solution phase (Gd-123ss) [5]. We have concluded from previous studies that (i) the addition of a small quantity of BaO_2 can suppress Gd/Ba substitution in the solid solution phase so that the superconducting properties can be improved and (ii) the addition of too much BaO_2, (above 4wt%, for example) leads to a reduction of the superconducting properties and growth rate. However, it had proved difficult to produce large single grains from a precursor with a high Ba content, so that the effects of adding Ba could not previously be understood fully. We also observed that the DTA traces of precursor powder containing added BaO_2 exhibit a single small peak below the melting temperature of Gd-123; BaO_2 addition can decrease the size of Gd-211 to less than 1 μm and tends to make their geometry more spherical. These results suggest that a new phase might form during processing either in the precursor powder or during single grain growth. As a result, a new processing route has been developed to enable growth of GdBCO single grains containing an increased amount of Ba, from Ba-rich precursors. Specifically, investigation of the Ba-rich corner of the $GdO_{1.5}$-BaO-CuO ternary phase diagram suggested that Gd-163 and Gd-143 are candidates for new type Ba-rich compound either for use as a precursor or as a new second phase within the superconducting bulk microstructure. As a result, Gd-163 and Gd-143 have been fabricated in both O_2 and in air for use as new type of Ba-rich precursor composition in this study. Both compounds were used separately in precursor powders to melt process single grains successfully. Here we report the influence of the addition of these new compounds on the microstructures and superconducting properties of GdBCO single grains grown by this new processing technique.

2. Experimental

Gd-163 and Gd-143 powders were synthesised by a conventional solid-state reaction technique using commercial Gd_2O_3, BaO_2 and CuO as starting materials. The mixed powders were calcined at 950 ^oC for 60 hours in flowing O_2 and air, respectively. The powders were then ground repeatedly until the X-ray diffraction (XRD) patterns confirmed the presence of only a single phase. The precursors for fabricating GdBCO single grains with starting compositions 70 wt% Gd-123 + (30-X) wt% Gd-211 + X wt% Gd-163 or Gd-143 + 0.1wt% Pt, where X = 5, 10, 15, and 30, were mixed and pressed uni-axially into pellets. A generic seed of melt-textured NdBCO with 1wt% of MgO [5] was placed on the top surface of the pellet prior to melt-processing (i.e. the cold-seeding method). The thermal profile used in this study is shown in Figure 1, where T_g is the growth temperature within the range 1040 ^oC to 1035 ^oC, depending on the peritectic decomposition temperature, T_p, of the different precursor compositions. T_p of each precursor powder was measured by differential thermal analysis (DTA). Single domains with different composition were annealed at 440°C-360^{o}C for 150 hours in flowing oxygen gas after completion of the melt process, as indicated in Figure 2.

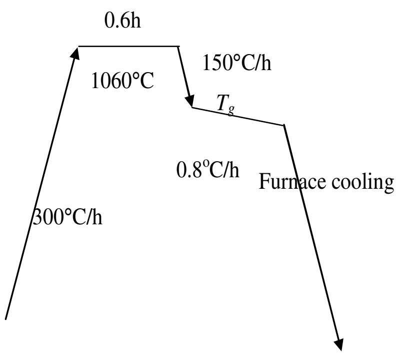

Figure 1 Processing profile for fabricating single grains with Gd-163 or Gd-143 addition

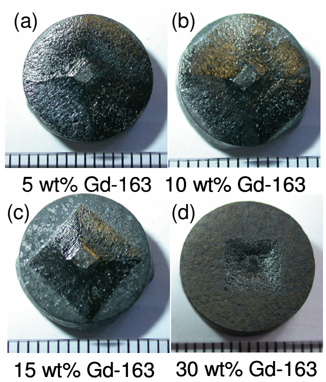

Figure 2 Photos of single grains with Gd-163 addition produced in air

The field trapping properties of the single grains were measured using a hall probe following field-cooling of the samples in an external magnetic field of 0.5 T. The two samples processed from 10wt% of Gd-163 and Gd-143, respectively were found to exhibit the highest trapped fields. These two samples were then cut into small specimens of approximately 1.0×1.2×0.5mm^3, as shown schematically in Figure 3 for measurement of their superconducting properties. Magnetisation measurements were performed using a commercial SQUID magnetometer to measure the superconducting transition temperature (T_c) and to estimate the critical current density (J_c). The Bean critical state model was used to calculate J_c from the measured magnetic hysteresis loops. Some of the processed single grains were crushed into powder for DTA and XRD measurements.

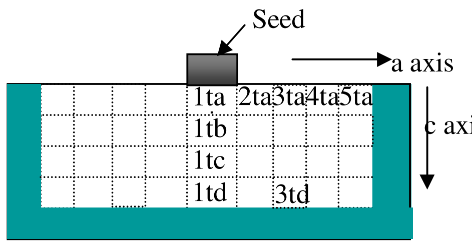

Figure 3 Schematic illustration of samples used for T_c, J_c measurements and micro-structural analysis

3. Results and discussion

3.1. Gd-163 and Gd-143 phases

Figure 4 shows the XRD patterns of Gd-163 and Gd-143 powders fabricated in flowing O_2. The XRD pattern of Gd-143 made in flowing O_2 is quite similar to that observed for the Gd-143 in an earlier study [6] except for trace of a Gd-211 phase. The XRD peaks for the Gd-163 prepared in O_2 could all be indexed to the calculated tetragonal phase of Gd-163 [7]. However, the Gd-163 synthesised in O_2 atmosphere is not stable at room temperature and atmosphere. It is observed that the powder changes its colour after being kept in air for a long time by reacting with air atmosphere. Therefore, the Gd-163 made in O_2 is not used in this study. The Gd-163 made in air shows the same XRD pattern as the Gd-143 made in O_2, but with broader peak width. The Gd-163 and Gd-143 powders used in the present single grain growth experiments are synthesised in air and in O_2 atmospheres, respectively.

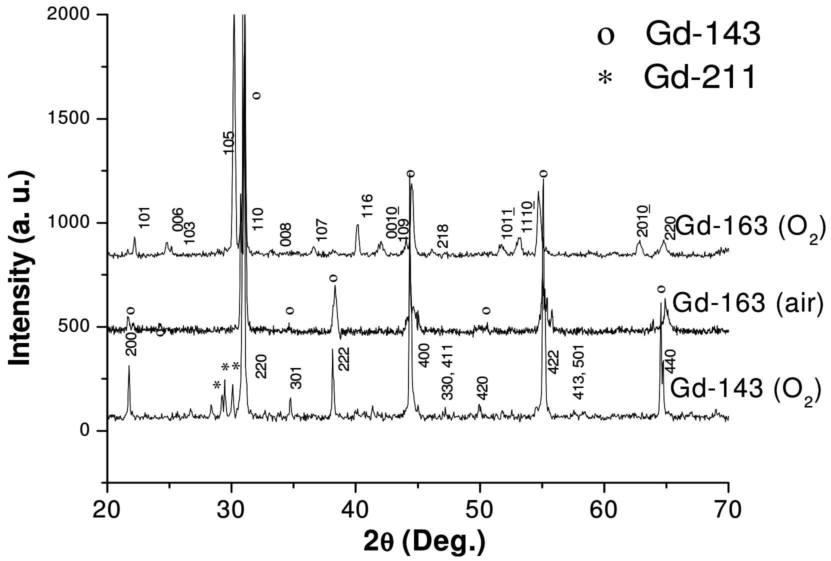

Figure 4 XRD patterns for Gd-143 and Gd-163 synthesised in flowing O_2 atmosphere and for Gd-163 in air.

3.2. Producing large single grains by a new processing route

Figure 2 shows photographs of the single grains produced by the cold seeding method with different concentrations of Gd-163. Each grain size is large and forms a complete single domain. The highest Gd-163 concentration of the precursor was 30wt%; it was not possible previously to fabricate a single grain with such high Ba concentration simply by adding BaO_2. The success of this new processing route may be attributed to the change in viscosity of the liquid phase during the single grain growth process due to the presence of the Gd-163 or Gd-143 phases, so that the mass transport between the liquid and the solid single grain becomes more rapid. This new processing route has enabled the effects of a high Ba concentration in the precursor powder, and hence bulk melt processed, superconducting single grains, to be investigated.

3.3. *Superconducting properties of the single grains prepared from Gd-163 and Gd-143 precursors*

Figure 5 shows the spatial variation of T_c along the *a*- and *c*-axies of single grains prepared from precursors containing 10wt% Gd-163 or 10wt% Gd-143. T_c of a small sample cut from directly beneath the seed position is the lowest and the transition width, ΔT_c, the highest for the sample fabricated without additional BaO_2 (Figure 5(d)). Such a variation in T_c as a function of position from the seed position is typical for LRE-BCO superconductors processed in air, since the solid solution level in the vicinity of the seed is the largest. It can be seen from Figs. 5(a), (b) and (c) that adding 10wt% of Gd-143 or 10wt% of Gd-163 to the precursor has the same effect on the spatial variation of T_c within the single grain as the addition of BaO_2 has on GdBCO. The measured value of T_c immediately under the seed increases and the onset T_cs within the whole single grains are almost constant (the variation is less than 0.5 K). T_c measurements for the single grain with higher Gd-143 or Gd-163 addition indicate a decrease in superconducting properties, as is observed with higher BaO_2 addition. Figure 6 shows J_c(B) for the sample containing 10 wt% Gd-163, which is similar to that of

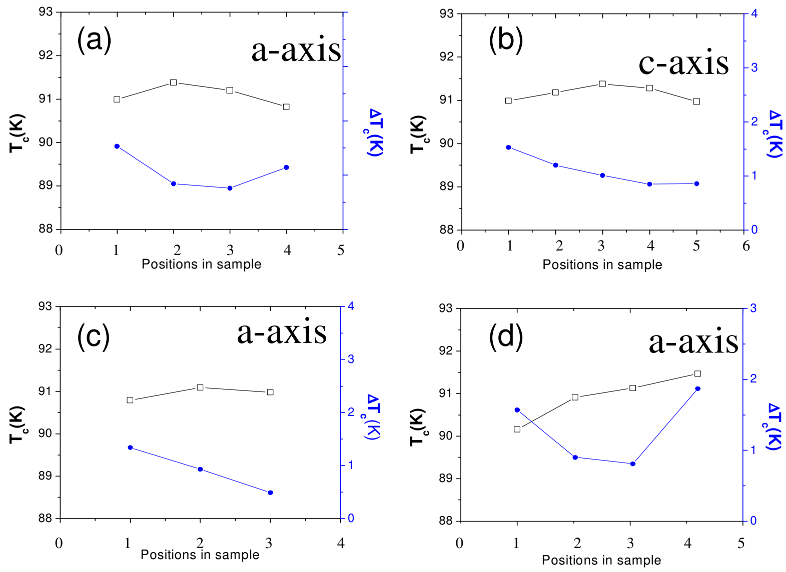

Figure 5 The spatial variation of T_c and ΔT_c for single grain along *a*- and *c*-axis, respectively fabricated with different starting composition. (a) 70 wt% Gd-123+ 20 wt% Gd-211 + 10wt% Gd-163 + 0.1wt% Pt in a-axis. (b) 70 wt% Gd-123+ 20 wt% Gd-211 + 10wt% Gd-163 + 0.1wt% Pt in c-axis. (c) The spatial variation of T_c and ΔT_c along a-axis for single grain fabricated with starting composition 70 wt% Gd-123+ 20 wt% Gd-211 + 10wt% Gd-143 + 0.1wt% Pt. (d) The spatial variation of T_c and ΔT_c along a-axis for sample without extra Ba addition (e.g 70 wt% Gd-123+ 30 wt% Gd-211 + 0.1 wt% Pt).

samples prepared with added BaO_2. These results indicate that an appropriate amount of Gd-163 or Gd-143 can be used to suppress the level of solid solution formation in GdBCO bulk superconductors.

3.4. Microstructures and lattice changes in processed GdBCO single grains

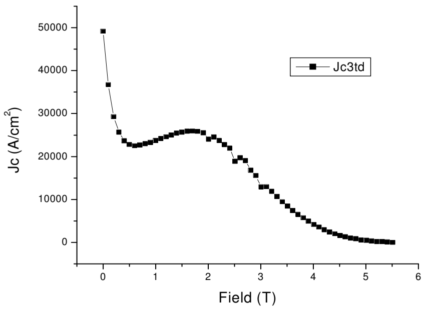

Figure 6 J_c of the sample containing 10 wt% Gd-163

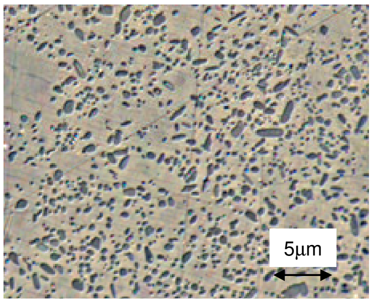

Figure 7 Microstructures of the single grain with 10 wt% Gd-163 in precursor

Figure 7 shows the microstructure of the single grain with 10 wt% Gd-163 added to the precursor. There are two distinct phases within the single grain microstructure; Gd-123 (background) and Gd-211 (small particles in irregular shape), which is typical for GdBCO single grains. Note that the presence of other second phases in the microstructure is not apparent at this level of magnification. Figure 8 shows the XRD pattern of a crushed single grain specimen with composition 70 wt% Gd-123+ 30 wt% Gd-163 + 0.1 wt% Pt in the precursor powder. All peaks in these data can be indexed as Gd-123, suggesting that the Gd-123 lattice in the single grain does not change as a result of a high Ba concentration in the precursor (the concentration of Gd-163 is as high as 30 wt%). Figure 9 shows the DTA trace of a ground single grain with 10wt% Gd-163 content in the precursor. The melting temperature is same as that of pure Gd-123. Figs. 7-9 suggest that the presence of Gd-163 within the precursor does not change the Gd-123 lattice of the fully processed single grain on the macro-scale, despite its relatively high initial concentration. The possibility of the formation of a second phase on the micro-scale has been studied further and will be reported elsewhere.

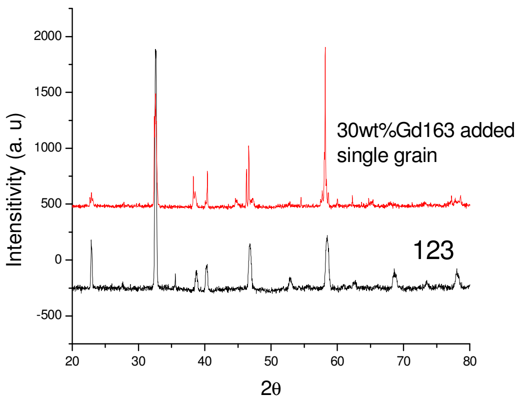

Figure 8 XRD pattern of a crushed single grain with composition 70wt%Gd123+ 30wt%Gd163 + 0.1wtPt

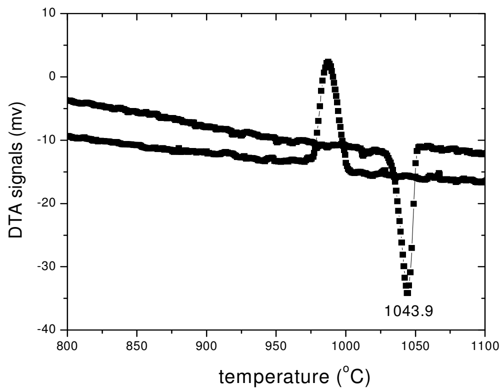

Figure 9 DTA curve of ground single grains with composition: 70 wt% Gd123 +20 wt% Gd-211 + 10 wt% Gd-163 163 + 0.1wt% Pt, measurements processed in air.

4. Conclusions

Gd-163 and Gd-143 powders have been synthesised successfully in O_2 and in air. A new processing route for the fabrication of GdBCO single grains has been investigated using Gd-163 processed in air or Gd-143 processed in O_2 as the source of a Ba-rich compound in the precursor powder. It has been shown that the new processing route can produce single grains with superconducting properties similar to those of GdBCO single grains prepared from precursor powder containing BaO_2. Furthermore, appropriate addition of Gd-163 or Gd-143 can suppress the formation of Gd/Ba solid solution and improve the superconducting properties of GdBCO single grains. Finally, it has been observed that processing large single grains with high Ba concentration becomes easier when Gd-163 is used in the precursor rather than BaO_2.

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