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Growth rate of YBCO single grains containing Y-2411(M)

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Abstract. Y-Ba-Cu-O (YBCO) single grains have the potential to generate large trapped magnetic fields for a variety of engineering applications, and research on the processing and properties of this material has attracted world-wide interest. In particular, the introduction of flux pinning centres to the large grain microstructure to improve its current density, J_c, and hence trapped field, has been investigated extensively over the past decade. $Y_2Ba_4CuMO_x$ [Y-2411(M)], where M = Nb, Ta, Mo, W, Ru, Zr, Bi and Ag, has been reported to form particularly effective flux pinning centres in YBCO due primarily to its ability to exist as nano-size inclusions in the superconducting phase matrix. However, the addition of the Y-2411(M) phase to the precursor composition complicates the melt-processing of single grains. We report an investigation of the growth rate of single YBCO grains containing Y-2411(Bi) phase inclusions and Y_2O_3. The superconducting properties of these large single grains have been measured specifically to investigate the effect of Y_2O_3 on broadening the growth window of these materials.

1. Introduction

YBCO large single grains have the ability to generate magnetic fields that are significantly greater than those produced by conventional permanent magnets, which makes them suitable for a variety of engineering applications. The focus of research in this area has been to increase the critical current, J_c, and hence to increase the associated trapped field of bulk samples. This is achieved most commonly by refining the size (to the nano-scale) and distribution of flux pinning centres in the bulk microstructure. Extensive research has been performed on reducing the particle size of the non-superconducting Y_2BaCuO_5 (Y-211), which forms intrinsically during the peritectic decomposition of the $YBa_2Cu_3O_7$ (Y-123) phase during melt processing [1][2][3]. These studies have concluded generally that J_c of YBCO single grains correlates inversely with the Y-211 particle size. In view of the tendency of the Y-211 phase to Ostwald ripening [4], however, there is a limitation on the extent to which these particles can be refined. Recently, a new $Y_2Ba_4CuMO_x$ [Y-2411(M)], where M = Nb, Ta, Mo, W, Ru, Zr, Bi and Ag, phase has been discovered by Hari Babu et al [5] to form particularly effective flux pinning centres in bulk (RE)BCO (where RE = rare earth element) due primarily to its ability to exist as stable, nano-size inclusions in the superconducting phase matrix. An increase in J_c by at least a factor of five has been reported for bulk nano-composites containing the Y-2411(M) phase compared to the undoped (RE)BCO system [5][6]. The addition of the Y-2411(M) phase to the

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precursor powder, however, presents a number of processing challenges for the fabrication of large single grains. For example, multi-grains may form under conditions of relatively low under-cooling during melt-processing, and the processing window for large grain formation can be reduced considerably by the presence of the Y-2411(M) phase. It was reported that the Y-123 + Y_2O_3 system has larger growth window compare to Y-123 + Y-211 system [7] and Yoeh's results suggested that adding nano-scale Y_2O_3 powder to the Y-123 system has the added advantage of yielding samples with significantly enhanced superconducting properties compared to the equivalent YBCO system fabricated from Y-123 + Y-211 [8].

In this study, we investigate the growth rate of four compositions of single YBCO grains containing Y-2411(Bi) phase inclusions, Y-211 and Y_2O_3. The aim of the study is to establish whether the presence of Y_2O_3 can aid the Y-123/Y2411(Bi) single grain growth process without degrading the superconducting properties of the fully processed sample.

2. Experimental

2.1. Synthesis of Y2411(Bi)

Y-2411(Bi) was synthesised via a conventional solid state reaction process from commercial Y_2O_3, BaO_2, CuO and Bi_2O_5 precursor powders. The mixed powders were calcined in air at temperatures between 880 °C and 1100 °C with several intermediate grinding stages until X-ray diffraction (XRD) confirmed the presence of a single phase. The XRD pattern for single phase Y-2411(Bi) is shown in Figure 1.

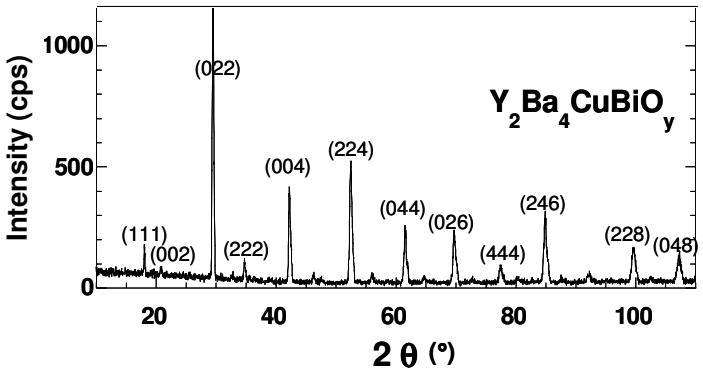

Figure 1. XRD pattern of the Y2411(Bi) powder

2.2. Fabrication of YBCO single grains in air under isothermal conditions

Four precursor powders were prepared of the following compositions: 1: [1.0 (mole) Y-123 + 0.25 (mole) Y-211]+ 0.1wt% Pt; 2: [1.0 (mole) Y-123 + 0.25 (mole) Y_2O_3] + 0.1wt% Pt; 3: [1.0 (mole) Y-123 + 0.25 (mole) Y_2O_3]+ 4wt%Y-2411(Bi) +0.1wt% Pt; 4: [1.0 (mole) Y-123 + 0.25 (mole) Y-211] + 4wt%Y-2411(Bi) + 0.1wt% Pt. Differential thermal analysis (DTA) (STA–780 SERIES) was carried out in flowing air on the precursor powders prior to melt processing by the top seeded melt growth (TSMG) technique at temperatures up to 1150 °C with a heating rate of 5 °C/min. The reacted powders were mixed thoroughly using a mortar and pestle and pressed uniaxially into cylindrical pellets of diameter 16 mm. A small (1.5 x 1.5 x 1 mm^3) NdBCO single grain seed was placed on the top of each pellet with its *ab*-plane in direct contact with the surface of the sample prior to melt processing (i.e. by the so-called cold seeding technique). Initially, four such samples were heated together rapidly in air in a box furnace to the melting temperature, T_m (1042 °C in this experiment), held for 1.2 hours to ensure complete peritectic decomposition of the Y-123 phase, cooled rapidly to T_{g1} (between 1004 °C and 987 °C) and held for various times between 4 and 50 hours. The processing

temperatures were chosen to provide a range of undercooling conditions prior to furnace cooling to room temperature in order to investigate the width of the growth window. T_{g1} was chosen according to the DTA analysis of the four precursor compositions.

2.3. Growth rate measurements of YBCO single grains processed under isothermal conditions

The size of the YBCO single grains grown by isothermal melt process is determined critically by the processing temperature and holding time. Each single grain (of varying size) was cut into two halves, with one half polished for measurement of the growth rate. Figure 2 illustrates schematically how the growth rate, defined as $L_{a/b}/t$ along the ab-plane, was measured (where $L_{a/b}$ is the length of the single grain along the a/b-axis and t is the growth time). The growth length for the limited number of single grains investigated was observed in this study to vary in proportion to processing time for a given level of undercooling, which is consistent with previous studies of the undoped YBCO system [9].

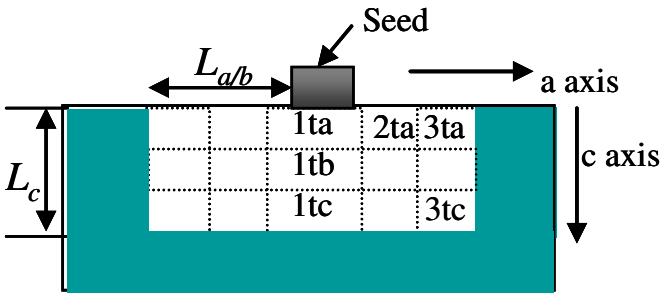

Figure 2. Illustration the measurement of growth rates and sample preparation for average Jc measurements

2.4. Measurements of superconducting properties

The superconducting transition temperature, T_c, and critical current density, J_c, were measured using a Quantum Design superconducting quantum interface device (SQUID). The extended Bean model [10] was used to calculate J_c from the width of the magnetic moment hysteresis loop. The average J_c (H) was calculated for 6 different small samples cut along a/b axis and c-axes as shown in figure 2.

3. Results and discussion

3.1. DTA results

Figure 3 shows the DTA analysis of commercial Y-123 powder and the precursor powders 1 to 4. It can be seen that the melting temperature decreases after the addition of Y-211 to Y-123 (precursor 1) compared to the pure Y-123 composition, but that the melting temperature of precursor 4 is almost the same as precursor 1 after adding 4wt% Y-2411(Bi). In addition, the melting temperature of precursor 2 decreases slightly after the addition of Y_2O_3 to pure Y-123 compared to the addition of Y-211. The melting temperature of precursor 4, however, is unchanged after adding Y-2411(Bi) to Y-123 + Y_2O_3. These results indicate that the addition of 4wt% Y-2411(Bi) does not change the melting temperature of the base precursor [i.e. that containing Y-2411(Bi)] independently of the system (Y-123 + Y-211 or Y-123 + Y_2O_3). It also can been seen that there is a small extra peak at about 920 °C for the curves 2 and 3, which are the DTA curves of the precursors containing Y_2O_3. These results are similar to that described in the literature [7], where it was mentioned that a liquid phase was formed at about 940 °C for the Y-123 + Y_2O_3 system and that this liquid phase had broadened the growth window of the system compared to that of the Y-123 + Y-211 system. This was because the liquid phases of Y-123 + Y_2O_3 system could exist in a wider temperature range from 940 °C to 1020 °C. The similar results from our study imply that adding Y_2O_3 to the systems (precursors 2 and 3) containing Y-2411(Bi) may

also broaden the growth window compared with that of the systems containing Y-211 (precursor 1 and 4).

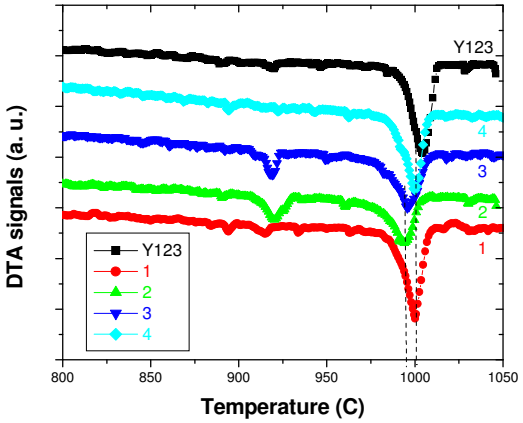

Figure 3. DTA results of the precursors 1 to 4

3.2. Growth rate

The temperature range for the measurement of growth rates was chosen to be between 1006 °C and 988 °C based on the results of the DTA. The higher temperature in this range was chosen to be several degrees higher than the highest melting temperature (i.e. the peak position of the DTA trace) of the precursor, which is about 998 °C. The growth temperature was reduced systematically until no grain growth was obtained under isothermal conditions without the formation of sub-grains. The holding time was chosen to be between 4 hours and 50 hours. It was determined from the growth length measurements of each single grain that the temperatures T_p at which single grains start to grow are 1005.0 °C, 1004.0 °C, 1002.0 °C and 1005.5 °C for the precursors 1 to 4, respectively. The measured growth rate for each precursor composition along the a/b axis of the grains, $R_{a/b}$, as a function of under-cooling ΔT (T_p–T_g) is shown in Figure 4(a). It can be seen roughly that the growth rate of precursors 1 and 4 and precursors 2 and 3 are similar and the growth rate for all compositions increases with increasing under-cooling.

In order to interpret the results more clearly and to determine the values of the parameters that best describe the data, analytical fits were made to the four growth rate curves. As is the case for YBCO and NdBCO, the relation between growth rate and under-cooling for GdBCO is observed to follow $R_{a/b} = \alpha(\Delta T)^{\beta}$ [where α and β are constants] for all compositions. The best fits were chosen by minimising the sum of the squares of the deviations between the theoretical and experimental data, as shown in Figure 4(b). The regulation values for the fitted curves 1 to 4 are in the range of 0.97 to 0.99. It can be seen clearly from Figure 4 that adding 4wt% Y-2411(Bi) to either the Y-123 + Y-211 or Y-123 + Y_2O_3 system does not change the growth rate significantly, although the maximum under-cooling width (i.e. the temperature range over which a single grain can grow without the formation of sub-grains) narrows for both Y-123 + Y-211 and Y-123 + Y_2O_3 precursor compositions. The maximum under-cooling widths for precursors 1 to 4 observed in this study are 14.0 °C, 17.0 °C, 13.0 °C and 12.5 °C respectively. The maximum under-cooling width for the Y-123 + Y_2O_3 + 4wt% Y-2411(Bi) composition is larger than that of Y-123 + Y-211 + 4wt%Y-2411(Bi).

The maximum under-cooling allowed under isothermal conditions is mainly associated with the growth window which is the temperature range at which the liquid phase is present. In the Y-123 + Y_2O_3 system, the liquid phase stays over a wider range (940°C ≤ T ≤ 1020°C) [7] than that of the Y-123 + Y-211 system. The results of the growth rate measurements suggest that adding Y_2O_3 has broadened the growth window of the YBCO system containing Y-2411(Bi). Furthermore, the growth

rate of the single grain fabricated from precursor 3 is also observed higher than of precursor 1 and 4. These results suggest that the presence of Y_2O_3 would aid the growth of the YBCO single grains containing Y-2411(Bi) significantly. The melt-processing of single grains at continues cooling condition (which is normal melt-processing method) has further confirmed that the single grains with composition 3 ([1.0 (mole) Y-123 + 0.25 (mole) Y_2O_3]+ 4wt%Y-2411(Bi) +0.1wt% Pt) are easy to growth as standard YBCO single grains although 4wt% Y-2411(Bi) is contained the composition.

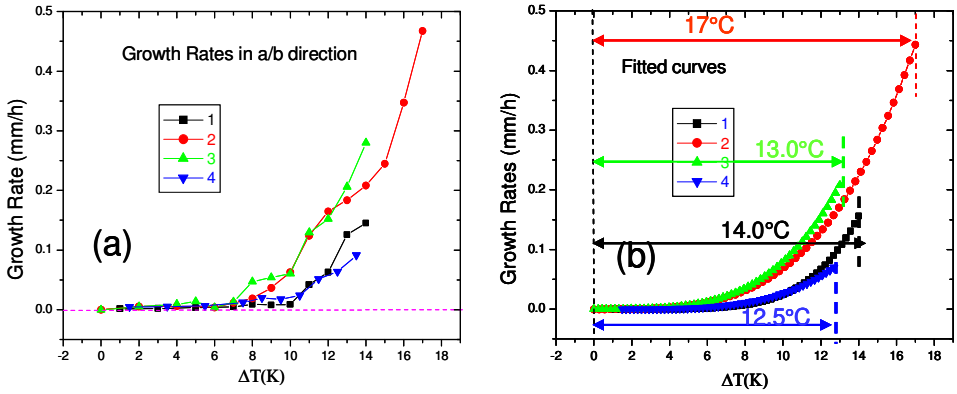

Figure 4. The relation between the growth rates of the single grains and undercooling

3.3. Superconducting properties

The presence of the Y-2411(Bi) phase does not change the T_c of the equivalent system. The T_c of single grains containing Y_2O_3 is reduced by approximately 0.7 °C compared to those containing Y-211. The reason for is unclear. It is generally believed that a reduction in T_c of this magnitude is not significant for the purposes of practical applications.

Figure 5 shows the average critical current density of the single grains fabricated from precursors 1 to 4. It can be seen from curves 1 and 4 that the presence of Y-2411(Bi) increases J_c in YBCO single grains for the Y-123 + Y-211 system, as reported previously [5][6]. The values of J_c for the Y-123 + Y_2O_3 system are naturally high, as is evident from curves 2 and 3, in good agreement with the results of ref. [9] (although nano-size Y_2O_3 was used in this research). The reason for the observed increase in J_c for the Y-123 + Y_2O_3 system may be associated with the higher volume fraction of smaller Y-211 particles, which form the main pinning centres in the (non Y-2411 containing) system same as reported in Ref. [9]. When Y-123 + Y_2O_3 is used as precursor, the Y-211 particles in the final single grain form from reactions on cooling; Y-123 + Y_2O_3 → Y-211 + CuO at 940°C [7]. It is this process that refines the Y-211 particle size, which accounts for the observed increased values of J_c.

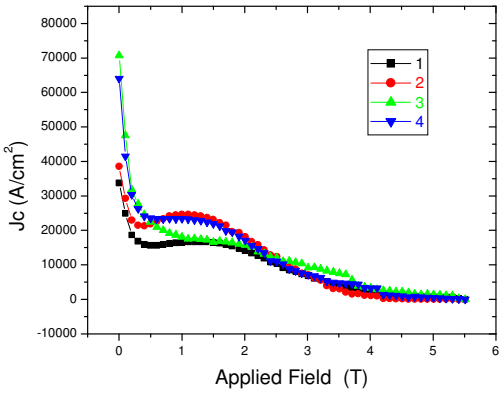

Figure 5. Average Jc values of single grains fabricated from precursor 1 to 4.

4. Conclusion

The results from the growth rates measurements indicate that the addition of Y_2O_3 to the YBCO precursor composition increases the growth rate and broadens the growth window of YBCO single grains containing Y-2411(Bi). The superconducting properties T_c and J_c of YBCO single grains are not degraded significantly after adding Y_2O_3 into the precursor containing Y-2411(Bi). Y_2O_3 can aid the Y-123/Y-2411(Bi) single grain growth process and single grains containing 4wt% Y-2411(Bi) can grow as easily as normal YBCO.

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