

Use of Sm-123 + Sm-211 Mixed-powder Buffers to Assist the Growth of SmBCO and ZrO₂-doped SmBCO Single Grain, Bulk Superconductors

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Abstract—In this paper single domain, bulk SmBCO samples have been fabricated successfully in air in a top-seeded melt growth (TSMG) process using a conventional chamber furnace, in addition to samples doped with ZrO₂. In order to improve the reliability of seeding, Sm-123+ Sm-211 mixed-powder buffers were used to increase the success of the SmBCO single grain growth process. SmBCO single grains of diameter as large as 20 mm and 10 mm in thickness with and without ZrO₂ were fabricated successfully using Sm-123 + Sm₂BaCuO₅ (Sm-211) mixed-powder buffer layers. The geometric configurations of the buffers were also optimized as part of this study to further increase the success of the single grain growth process. Superconducting properties of J_c and T_c of the specimens under the buffer layer have also been investigated.

Index Terms—buffers, doping effects, high-temperature superconductors, SmBCO, top seeded melt growth

I. INTRODUCTION

SM-BA-CU-O (SmBCO) high temperature superconductors (HTS) have the potential to exhibit significantly higher critical current densities, J_{cs} , and higher irreversibility fields than Y-Ba-Cu-O (YBCO) [1]-[3]. The top-seeded melt growth (TSMG) process with Mg-doped NdBCO generic seeds (MgO-NdBCO) is used commonly to fabricate SmBCO single grains with controlled orientation. However, due primarily to the high melting temperature, rapid growth rate and the need to process the material under reduced oxygen partial pressure to inhibit the substitution of Sm on the Ba site in the superconducting SmBa₂Cu₃O_{7- δ} (Sm-123) phase [4], it is extremely difficult to grow large SmBCO single grains in air with good superconducting properties, even without the addition of dopants.

We have reported the development of a MgO-NdBCO generic seed [5] for melt-processing (LRE) BCO bulk superconductors, where LRE is a light rare earth element such as Nd, Sm, Eu or Gd, in air by TSMG. The generic seed has a

higher melting point and a similar crystal structure to all bulk superconductors in the (LRE) BCO family, although the difference in melting temperature between the seed and the SmBCO precursor powders is relatively small. In principle, therefore, Mg-doped, melt textured NdBCO can be used as a generic seed to grow any (LRE) BCO single grain bulk superconductor in air to overcome the high melting temperature of the bulk [6]. However, although MgO-NdBCO generic seeds can achieve better growth orientation, it is difficult to choose an appropriate seed for the synthesis of bulk single grains, like other seeds. Essentially, the seed should not be too thin or too small, and even seeds that appear flawless optically still often fail to nucleate the single grain growth process. As a result, the choice of a seed requires skill and is potentially time-consuming. Furthermore, the cause of growth failure due to poor characteristics of the seed is not usually obvious, and inevitably leads to waste material. Therefore, improving the reliability of the seeding process is an urgent requirement of the TSMG process.

Several attempts to grow melt-processed SmBCO bulk superconductors have been reported in the literature [6]-[8]. At the same time, attempts to improve the superconducting properties of SmBCO have been reported, including the addition of Sm-2411 as a new kind of pinning center [9] and SmBa₂Cu_{2.67}Al_{0.33}O_{6+ δ} as a novel dopant [10]. However, these approaches only improve the superconducting properties of SmBCO to a limited extent and do not generally enable scale-up of production, which is critical for the development of practical applications. Other approaches such as the addition of Ag to improve the mechanical properties of SmBCO have also been used to try to scale up the production process [11]-[13], although none of these studies have been able to yield larger grains in air in a TSMG process using a standard chamber furnace. Also, several groups have observed successful growth of YBCO, GdBCO and NdBCO bulks using buffered seeds with different buffer layer compositions [14]-[17]. Yet, fewer attempts have been reported to apply such a technique to SmBCO system. No research on large scale production has succeeded and the reliability of the buffers is not certain [18].

This paper addresses directly the above challenges of seeded melt processing and large grain growth via the use of Sm-123 + Sm₂BaCuO₅ (Sm-211) mixed-powder buffers to produce single domain, ZrO₂ doped and undoped bulk SmBCO samples as large as 20 mm in diameter and 10 mm in

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thickness in air by TSMG using conventional processing equipment. In addition, MgO-NdBCO generic seeds that are small or thin have been used with a much lower single grain failure rate via optimization of the geometric configurations of the buffer layers.

II. EXPERIMENTAL

A. Production of SmBCO single grains in air by cold seeding

1) SmBCO bulk pre-form preparation

Precursor powders were prepared using commercially available Sm-123 (TOSHIMA, average particle size: 2-3 μm), Sm-211 (TOSHIMA, average particle size: 1-2 μm), BaO₂ (ALDRICH, purity 95 %) and CeO₂ (Alfa Aesar, purity 99.9 %) powders. A motorized pestle and mortar was used to mix thoroughly powders of composition (75 wt% Sm-123 + 25 wt% Sm-211) + 2 wt% BaO₂ + 1 wt% CeO₂, prior to being pressed uniaxially under a pressure of 1.5 tons into a green pre-form (pellet-like) with diameters of 20 mm and 25 mm and thicknesses of 9 mm and 12.5 mm (each pellet shrinks to about 80 % of its original size after TSMG, corresponding to as processed dimensions of 16 mm and 20 mm diameter and 7 mm and 10 mm thickness). Doped green pellets were also prepared by adding 1 mol% and 2 mol% ZrO₂ (commercial ZrO₂ supplied by Alfa Aesar, purity 99 %) into precursor powders. The powder mixing procedure was largely similar to that of the SmBCO precursor pellets, except for the presence of dopant in the starting powders. Therefore, the doped samples had the same overall volume and weight as the undoped SmBCO green pellets.

2) Buffer preparation

Buffers with three different compositions, as summarized in Table 1, were prepared in this research using commercially available Sm-123 (TOSHIMA, average particle size: 2-3 μm), Sm-211(TOSHIMA, average particle size: 1-2 μm), BaO₂ (ALDRICH, purity 95 %) and CeO₂ (Alfa Aesar, purity 99.9 %) powders.

TABLE I
DIFFERENT COMPOSITIONS OF MIXED-POWDER BUFFERS

	Sm-123	Sm-211	BaO ₂	CeO ₂
Sm-211 buffers	×	√	×	×
SmBCO buffers	√	√	√	√
Sm-123 + Sm-211 mixed-powder buffers	√	√	×	×

A motorized pestle and mortar was again used to mix thoroughly powders of the different compositions, prior to being pressed uniaxially under a pressure of 0.5 tons into a green pre-form (pellet-like) with the dimensions listed in Table 2.

TABLE II
GEOMETRIC CONFIGURATIONS OF DIFFERENT BUFFERS

Sample No.	Diameter / mm	Thickness / mm
1	5	5
2	3	1.5
3	3	3
4	3	4.5

3) Top-seeded melt growth (TSMG) process

The TSMG technique with a modified temperature profile was used to fabricate large, single grain SmBCO superconductors. A MgO-NdBCO generic seed was placed with its a/b-plane in direct contact with the top of the as-prepared buffer and the arrangement placed at the centre of the upper surface of the sample, as shown in Fig. 1, to yield the required grain orientation.

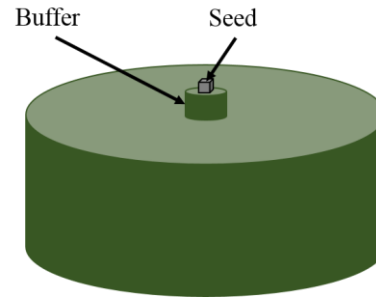


Fig. 1. Illustration of the bulk pre-form with a MgO-NdBCO generic seed and a buffer

The heating profile used in this research is shown in Fig. 2. The appropriate values of seed melting point and grain nucleation temperature, crystallization and the optimum final growth temperature, T_m , T_s , T_{g1} and T_{g2} , were determined from differential thermal analysis (DTA). The pellet was initially heated slowly at 50 °C·h⁻¹ to 200 °C to stabilize the furnace temperature, then more rapidly at a rate of 200 °C·h⁻¹ to 900 °C. The temperature was then raised slowly to 1087 °C and held for 0.5 hour to ensure complete decomposition of the precursor pellet. The partially molten sample was then cooled at 75 °C·h⁻¹ to its seeding temperature, followed by further cooling to its crystallization temperature T_{g1} (c.a. 1075 °C), then cooled slowly to T_{g2} (c.a. 1067 °C) at a rate of 1 °C·h⁻¹, followed by another slower cooling stage to 1041 °C at the rate of 0.8 °C·h⁻¹. Finally, the sample was furnace cooled to room temperature at 150 °C·h⁻¹.

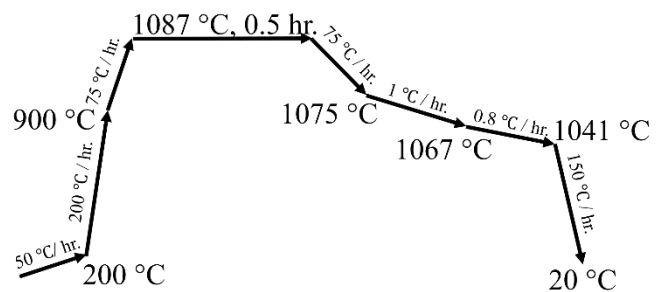


Fig. 2. Schematic illustration of the modified TSMG thermal process for SmBCO growth

A low temperature post-annealing process was developed to overcome the acute sensitivity of the superconducting

properties to the extent of Sm/Ba substitution in the SmBCO and ZrO₂-doped SmBCO single grains, based on the results of a large number of measurements, including T_c , DTA and XRD [9]. Samples of all compositions were annealed under an atmosphere of 0.1 % O₂ in Ar at 850 °C and were then oxygenated at 380 °C for seven days to drive the non-superconducting, tetragonal Sm-123 phase to the desired orthorhombic, superconducting phase.

B. Characterisation

1) Differential thermal analysis (DTA)

The melting (T_m), seeding (T_s), crystallization (T_{g1}) and final growth (T_{g2}) temperatures were measured using differential thermal analysis (DTA). Pellets of the same diameter and thickness of 3 mm with the composition of SmBCO, 1 mol% ZrO₂-doped SmBCO, 2 mol% ZrO₂-doped SmBCO and several small MgO-NdBCO generic seeds were prepared for DTA measurements. In each case, a small, generic seed was placed at the center of the top surface of the pellet to simulate the TSMG process at elevated temperature.

2) Microstructures of bulk single grains

Optical microscopy was used to examine the size and distribution of Sm-211 particles and the aggregation of CeO₂ in the as-grown SmBCO single grains, along both the a/b- and c-axes. The as-prepared superconducting pellets were cut into two halves along the c-axis through the seed and the exposed cross-section was polished sequentially using 120, 220, 320, 800, 1000, 1200, 2400 grit SiC papers. Further polishing was achieved by using 1 μm diamond spray. A Nikon Eclipse ME600 optical microscope was used to observe the section microstructures.

3) Measurements of J_c and T_c of the bulk specimens

The samples were cut into slices across their centre, with each slice being cut into smaller sub-specimens, as shown schematically in Fig. 3. The spatial changes in J_c and T_c of the sub-specimens with proximity to the seed were measured using a superconducting quantum interference device (SQUID) MPMS XL magnetometer. A field of 20 Oe was applied to the samples after zero-field-cooling prior to the measurement of T_c . The extended Bean model was used to calculate J_c from the measured magnetic hysteresis loops (M-H loops). J_c could be calculated using the formula $J_c = 20\Delta m / (a \times b \times c) / (a \times (1 - a/3b))$ (A·cm⁻²), where Δm (emu) represents the difference magnetic moment observed in the M-H loops for increasing and decreasing field cycles with the applied field perpendicular to the ab-plane, and a, b and c are the dimensions of the samples in the a, b, c directions, respectively, (where a < b), measured in centimetres.

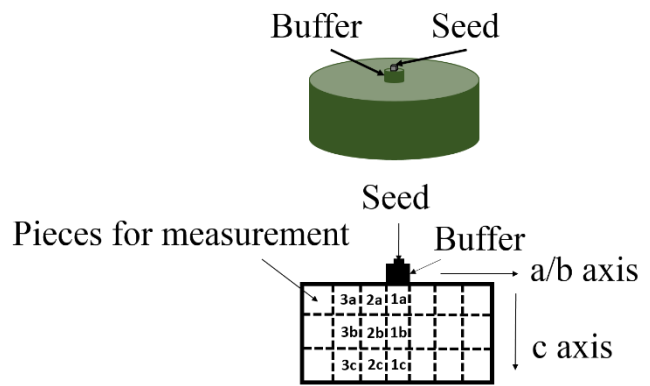


Fig. 3. How each single grain was cut following melt processing and illustration of the position of the sub-specimens within the parent bulk used for measuring J_c and T_c .

III. RESULTS AND DISCUSSION

Fig. 4 shows DTA signals of SmBCO, 1 mol% ZrO₂-doped SmBCO, 2 mol% ZrO₂-doped SmBCO and MgO-NdBCO generic seeds for purposes of comparison (for a more accurate comparison, T_m is discussed using peak-temperature from DTA as a benchmark). It can be seen from Fig. 4 that the melting temperature of the MgO-NdBCO generic seeds is around 1102.7 °C, which is roughly 31 °C higher than that of the SmBCO sample (approximately 1071.8 °C). In order to determine the heating profile, T_m should be set higher than the melting temperature of the powders to ensure thorough powder melting, while, at the same time, lower than the melting temperature of the generic seed to guarantee the integrity of the seed during melt processing. This principle can be applied to the TSMG processing of all (LRE) BCO bulk superconductors. T_m has even been set at around 50 °C higher than the melting temperature of YBCO precursor powders in previous studies [19], which made it easier to synthesize YBCO single grains. However, due to the higher melting temperature of the SmBCO precursor powders, T_m that is only 10-20 °C higher than the SmBCO melting temperature was used in this research, which is another reason why SmBCO is more difficult to grow than other (LRE) BCO bulk superconductors. In addition, the melting point of the doped SmBCO sample (around 1070.0 °C) was observed to be similar to that of the standard sample (around 1071.8 °C), suggesting that, in order to grow a ZrO₂-doped SmBCO single grain, the heating profile should be adjusted accordingly.

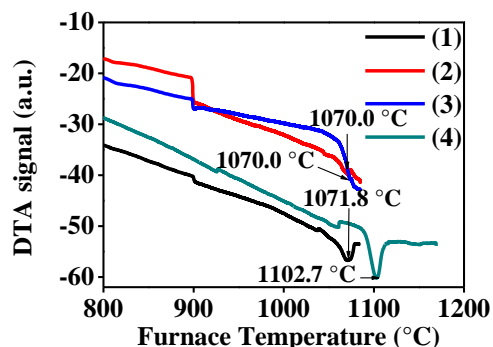


Fig. 4. DTA traces of (1) SmBCO, (2) 1 mol% ZrO₂-doped SmBCO, (3) 2 mol% ZrO₂-doped SmBCO and (4) MgO-NdBCO generic seeds

Fig. 5(a) shows a photograph of the top surface of a SmBCO sample (10 g, 16 mm in diameter) grown successfully using this process, which is evident from the presence of the four orthogonal growth sector boundaries. Unfortunately, the attempt to melt process samples of 20 g and diameter 20 mm failed due to the poor shape of the generic seeds, which were cut artificially from the parent bulk sample. Buffers of 5 mm in both diameter and thickness with compositions of Sm-211 and SmBCO were used subsequently to grow SmBCO samples of a similar weight and dimensions. Photographs of the top surfaces of these samples are shown in Figs. 5(b) and (c). It can be seen clearly that neither Sm-211 nor SmBCO buffers can be used to fabricate larger SmBCO grains successfully.

Further trials were made using Sm-123 + Sm-211 mixed powders as buffers to fabricate SmBCO single grains of mass 10 g and diameter 16 mm using buffers of diameter 5 mm and 3 mm. Photographs of both successfully-grown samples are shown in Figs. 5(d) and (e).

SmBCO samples of mass 20 g and diameter 20 mm were subsequently synthesized successfully by using a 3 mm Sm-123 + Sm-211 mixed-powder buffer, as shown in Fig. 5 (f). The optimum geometric configuration of the buffer layer was determined by preparing three different sizes of sample (listed in Table 1 as No. 2 (g), No.3 (h) and No.4 (i)). Samples grown using these buffers are shown in Figs. 5(g), (h) and (i). Only (h) is grown in the form of a SmBCO single grain, with (g) and (i) containing either satellite grains or double facet lines. It can be concluded from this figure that buffers with an aspect ratio of 1 (*i.e.* buffer diameter = buffer thickness) are the most advantageous for growing larger, single grain SmBCO bulk superconductors.

Using the optimized composition and geometric configurations of the buffer layer (75 wt% Sm-123 + 25 wt% Sm-211, d=3 mm, t=3 mm), SmBCO doped with 1 mol% and 2 mol% ZrO₂ (mass 20 g, diameter 20 mm) were also grown successfully by slight adjustment of the heating profile. Photographs of these samples are shown in Figs. 5 (j) and (k). These results demonstrate clearly that the use of optimized buffer layers have the potential to overcome the seed-based difficulties for the growth of larger SmBCO superconductors.

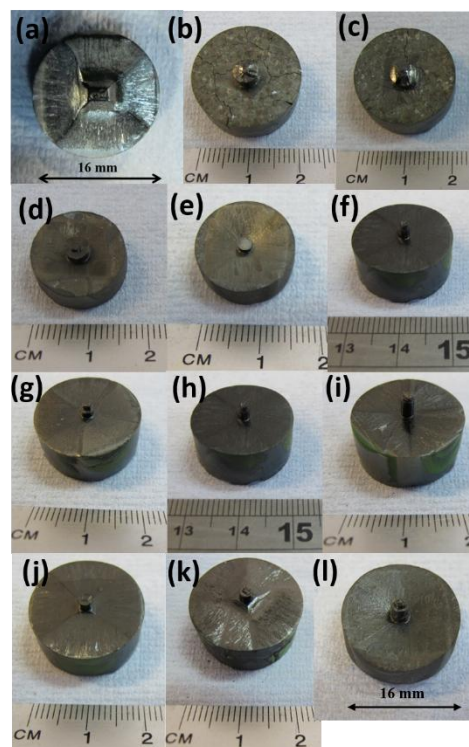


Fig. 5. (a) Single grain SmBCO fabricated successfully in this study, SmBCO fabricated using different buffers of composition (b) Sm-211, (c) SmBCO, geometry (d) 5 mm in diameter, (e) 3 mm in diameter, (f) SmBCO (20 g, 20 mm diameter) fabricated using a 3 mm Sm-123 + Sm-211 mixed powder buffer, SmBCO (20 g, 20 mm diameter) fabricated using three different sizes of Sm-123 + Sm-211 mixed powders as buffers, (g) d=3 mm, t=1.5 mm, (h) d=3 mm, t=3 mm, (i) d=3 mm, t=4.5 mm; doped SmBCO (20 g, 20 mm) fabricated using Sm-123 + Sm-211 mixed powders as buffers: (j) 1 mol% ZrO₂-doped SmBCO, (k) 2 mol% ZrO₂-doped SmBCO; (l) Bulk single grain with the same composition of Sm-123 + Sm-211 mixed-powder buffer fabricated in this research.

In order to investigate further the properties of the buffer layer, standard SmBCO and 1 mol% ZrO₂-doped SmBCO samples were prepared and examined using optical microscopy. Previous research indicates that Y-211 particles tend to distribute throughout an as-grown single grain bulk sample [19]. However, examination of the sample microstructures in this study shows that there are no obvious differences between SmBCO and 1 mol% ZrO₂-doped SmBCO. The Sm-211 distribution and particle size are dispersed uniformly throughout the whole sample, as shown in the micrographs in Fig. 6, taken 2 mm from the seed along the c-axis. In addition, there is no obvious aggregation of the CeO₂ phase, which could be one reason why SmBCO samples are difficult to grow in large sizes (the role of CeO₂ is mainly to inhibit Sm-211 particles from coarsening). Aggregation of CeO₂ particles would therefore yield coarser Sm-211 particles, which would impede single grain growth. Bulk single grains with the same composition of Sm-123 + Sm-211 mixed-powder buffers were also grown successfully using the optimized buffer layer, as shown in Fig. 5(l). This suggests that this layer effectively forms a seed that overcomes the lattice mismatch between MgO-NdBCO generic seeds and the SmBCO bulk single grain. This suggests, therefore, that the

optimized buffers could also be used to fabricate SmBCO containing CeO₂ and other dopants, indicating that buffers are relatively insensitive to the presence of impurities inside the precursor pellets compared to MgO-NdBCO generic seeds.

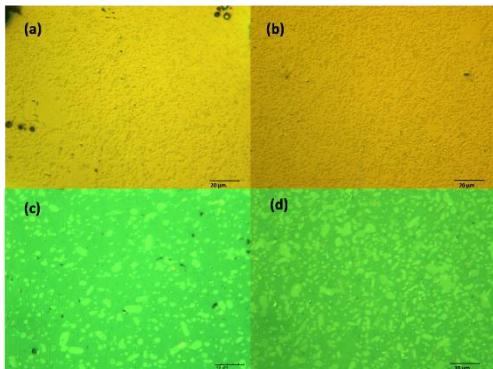


Fig. 6. Micrographs showing the Sm-211 distribution and particle size in SmBCO and 1 mol% ZrO₂-doped SmBCO: with the magnification of 250 times: (a) SmBCO; (b) 1 mol% ZrO₂-doped SmBCO; with the magnification of 500 times: (c) SmBCO; (d) 1 mol% ZrO₂-doped SmBCO

Superconducting properties (J_c and T_c) of the 1a position of the as-prepared samples are summarized in Fig. 7 and Fig. 8, respectively. The samples in Fig. 7 and Fig. 8 are SmBCO fabricated without a buffer layer, undoped SmBCO fabricated using a 3 mm Sm-123 + Sm-211 mixed powder buffer, 1 mol% and 2 mol% ZrO₂-doped SmBCO fabricated using a 3 mm Sm-123 + Sm-211 mixed powder buffer. Two aspects of the J_c data in Fig. 7 were compared: the ‘peak effect’ and irreversibility field. Both ZrO₂-doped SmBCO samples have the highest values. Samples with or without buffer layers showed similar J_c values as well. These results showed that buffers can not only stop contaminations from the Mg-doped seed, but also improve the reliability of seeding. The onset T_c in Fig. 8 for all four specimens is about 93 K, with a relatively sharp transition width of less than 1 K. This shows that ZrO₂ is an effective dopant. Also, the results indicate the success of the single grain fabrication process with buffer layers.

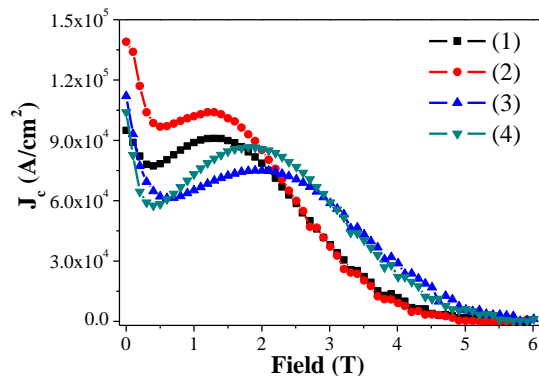


Fig. 7. Comparison of J_c among the 1a position of (1) SmBCO fabricated without buffer layer, samples fabricated using a 3 mm Sm-123 + Sm-211 mixed powder buffer: (2) SmBCO, (3) 1 mol% ZrO₂-doped SmBCO and (4) 2 mol% ZrO₂-doped SmBCO.

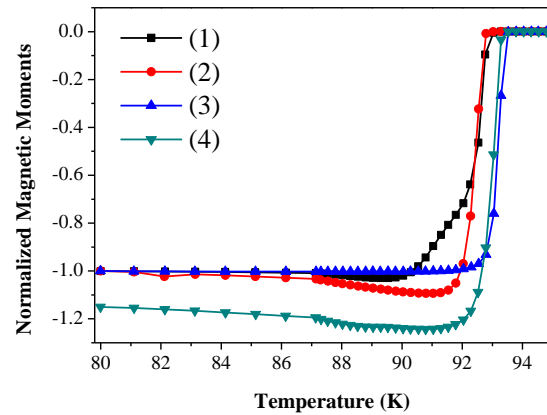


Fig. 8. Comparison of T_c among the 1a position of (1) SmBCO fabricated without buffer layer, samples fabricated using a 3 mm Sm-123 + Sm-211 mixed powder buffer: (2) SmBCO, (3) 1 mol% ZrO₂-doped SmBCO and (4) 2 mol% ZrO₂-doped SmBCO.

IV. CONCLUSIONS

Large SmBCO and ZrO₂-doped SmBCO single grains up to 20 mm in diameter and 10 mm in thickness with starting compositions of (75 wt% Sm-123 + 25 wt% Sm-211) + 2 wt% BaO₂ + 1 wt% CeO₂ + 1 mol%, 2 mol% ZrO₂ (added when doped samples were synthesized) have been fabricated successfully by TSMG in air using Mg-doped generic seeds in a conventional chamber furnace. The use of buffer layers make the choice of seed crystal less critical to the success of the growth process, aid the uniform distribution of Sm-211 particles throughout the single grain sample, overcome the lattice mismatch between MgO-NdBCO generic seeds and the SmBCO bulk single grains and are relatively tolerant to the presence of impurities in the precursor pellets compared to MgO-NdBCO generic seeds. Finally, buffer layers with an aspect ratio of 1, where the thickness is equal to the diameter (*i.e.* d: t=1:1) represents an optimum geometry for SmBCO single grain growth, as has been demonstrated for buffers with d = t = 3 mm.

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