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A new seeding technique for the reliable fabrication of large, SmBCO single grains containing silver using top seeded melt growth

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Abstract

Silver (Ag) is an established additive for improving the mechanical properties of single grain, (RE)Ba₂Cu₃O_{7-δ} [(RE)BCO, RE = Sm, Gd and Y] bulk superconductors. The presence of Ag in the (RE)BCO bulk composition, however, typically reduces the melting temperature of the single crystal seed in the top seeded melt growth (TSMG) process, which complicates significantly the controlled nucleation and subsequent epitaxial growth of a single grain, which is essential for high field engineering applications. The reduced reliability of the seeding process in the presence of Ag is particularly acute for the SmBCO system, since the melting temperature of SmBCO is very close to that of the generic NdBCO(MgO) seed. SmBCO has a high superconducting transition temperature, T_c , and exhibits the most pronounced 'peak' effect at higher magnetic field of all materials in the family of (RE)BCO bulk superconductors and, therefore, has the greatest potential for use in practical applications (compared to GdBCO and YBCO, in particular). Development of an effective seeding process, therefore, is one of the major challenges of the TSMG process for the growth of large, high quantity single grain superconductors. In this paper, we report a novel technique that involves introducing a buffer layer between the seed crystal and the precursor pellet, primarily to inhibit the diffusion of Ag from the green body to the seed during melt processing in order to prevent the melting of the seed. The success rate of the seeding process using this technique is 100% for relatively small batches of samples. The superconducting properties, critical temperature, T_c , critical current density, J_c and trapped fields, of the single grains fabricated using the buffers are reported and the microstructures in the vicinity of the buffer of single grains fabricated by the modified technique are analysed to understand further the effects of buffers on the growth process of these technologically important materials.

Keywords: buffer, SmBCO–Ag single grains, reliable seeding, silver, superconducting properties

(Some figures may appear in colour only in the online journal)

1. Introduction

Bulk, single grain superconductors in the (RE)Ba₂Cu₃O_{7-δ} [(RE)BCO, RE = Sm, Gd and Y] have considerable potential for practical applications due to their ability to trap magnetic fields up to ten times higher than the fields available using

conventional permanent magnets [1]. Unfortunately, however, the oxide nature of (RE)BCO single grains means they tend to form brittle compounds and exhibit mechanical properties that are characteristic of ceramic materials. This is particularly significant given that the tensile forces associated with a high, trapped magnetic field can be hundreds of tonnes,



which makes single grain bulk superconductors extremely susceptible to mechanical failure by fractures initiating from defects, pores and cracks that pre-exist in as-processed single grains. As a result, Ag has been used for some time to improve the mechanical properties of GdBCO single grains by in-filling the pores in the single grain microstructure generated during peritectic solidification [2–4]. However, the presence of Ag in the precursor pellet reduces the melting temperature of the single crystal seed in the top seeded melt growth (TSMG) process, which complicates significantly the controlled nucleation and subsequent epitaxial growth of a single grain, which is essential for high field applications. SmBCO has a high superconducting transition temperature, T_c , and exhibits the most pronounced ‘peak’ effect at higher magnetic field of all materials in the family of (RE)BCO bulk superconductors [5, 6] and, therefore, has the greatest potential for use in practical applications (compared to GdBCO and YBCO, in particular). The reliability of the seeding process reduces significantly when Ag is introduced to the SmBCO system, in particular, where the melting temperature of the bulk composition is very close to that of the generic, NdBCO(MgO) seed. Failure of the seed is a direct cause of failure of the single grain growth process. Therefore, the development of an effective and reliable seeding technique is one of the greatest challenges in fabricating large quantities of SmBCO–Ag single grains. As a result, it is extremely important to increase the success rate of the TSMG process to batch-produce samples for commercial applications of these technologically important materials, and particularly so given the increasing cost and limited availability of rare-earth elements. In this paper, we report a novel technique that involves introducing a buffer layer between the seed and the precursor pellet that forms a physical barrier to inhibit the diffusion of Ag from the green body to the seed during melt processing in order to prevent the melting of the seed. The use of the buffer solves completely the issue of the seed melting, and increases the success rate of the seeding process to 100% of the samples fabricated in small batches by this process to date. Batch processed, single grains fabricated by this technique are presented, their microstructures in the vicinity of the buffer are analysed and their superconducting properties, including T_c , J_c and trapped fields, are measured in order to demonstrate the positive effects of the buffers. A number of reasons for the apparent success of the novel seeding process are proposed.

2. Experimental

2.1. A longstanding problem with seeded growth of SmBCO–Ag

Figure 1 shows five SmBCO–Ag samples that were grown together as a batch in a box furnace using a common TSMG process in an air atmosphere. Generic, NdBCO(MgO) seeds [7, 8] were used to nucleate grain growth for each sample, as shown in figure 1, from which it can be seen that the seed was used successfully to grow single grain SmBCO–Ag samples



Figure 1. SmBCO–Ag samples demonstrating the failure of the seed (sample 2).

(labelled 1, 3, 4, and 5). Sample 2 (top left sample), however, failed to grow fully into a single grain due to a problem with the seed, and consists of multi-grains. The resulting seeding success rate of 80% achieved in this experiment, which is typical for a standard SmBCO batch process of this type, is not good enough for the large-scale fabrication of this material for commercial applications. SmBCO has a higher melting temperature than YBCO and GdBCO (the three most popular and potentially applicable (RE)BCO systems and is therefore the closest to the melting temperature of the seed used in the melt-process. Adding Ag to SmBCO decreases the melting temperature of the seed by about 40 °C, which makes the melt processing of SmBCO–Ag single grains extremely challenging. The maximum temperature during processing, T_m , and holding time at T_m were 1071 °C and 10 min, respectively. This value of T_m corresponds roughly to the melting temperature of undoped SmBCO, which is necessarily low to avoid the failure of the seed (T_m is usually required to be about 35 °C higher than the melting temperature of the SmBCO–Ag system to be seeded).

2.2. A new approach to seeded melt growth

Higher T_m and longer holding times are preferred in the TSMG of (RE)BCO to achieve complete peritectic decomposition of the superconducting $\text{REBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (RE-123) phase to the $\text{RE}_2\text{BaCuO}_5$ (RE-211) and $\text{Ba}_3\text{Cu}_5\text{O}_8$ liquid phases (where RE=Sm, Gd and Y). However, the seed crystal is much more likely to fail when a higher T_m and longer holding time is used for melt processing when Ag is present in the mixed precursor powder. A material buffer between the pellet and seed to isolate the seed from Ag in the precursor pellet during processing was therefore used in an attempt to maintain a relatively high decomposition temperature of the seed, at least during the nucleation and initial growth phase of the SmBCO/Ag single grain. Figure 2 shows three arrangements of the SmBCO–Ag green pellet prior to melt processing, each using a generic MgO–NdBCO seed (G-seed). In each case, the composition of the SmBCO–Ag pellet was (75 wt% Sm-123 + 25 wt% Sm-211) + 1 wt% CeO_2 + 2 wt % BaO_2 + 10 wt % AgO_2 . The commercial precursor powders had a purity of 99.9%. In total six pellets, each of mass 10 g, were prepared, again as shown in figure 2,

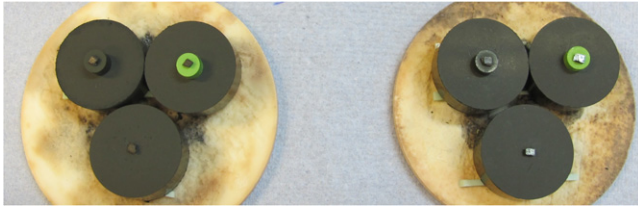


Figure 2. Two sets of three, different seed-precursor pellet arrangements. The Sm-211 (green phase) buffer is clear from the figure.

with three types of seed-precursor pellet arrangements and two buffer pellets investigated purposefully for this study. The first type of buffer was of composition 75 wt% Sm-123 + 25 wt% Sm-211, with a weight of 0.3 g and diameter of 5 mm. The second buffer consisted only of Sm-211, but, again, had a weight of 0.3 g and a diameter 5 mm. The third arrangement consisted only of generic seed placed in direct contact with the top surface of the pellet. The same heating profile was used for all six samples, with a T_m and holding time at T_m of 1071 °C and 20 min, respectively. The temperature was measured using a thermocouple located at the back of the furnace chamber, positioned approximately 5 cm away from each sample.

2.3. Measurements of the superconducting properties, T_c , J_c and trapped fields, of the SmBCO–Ag single grains batch-fabricated using buffers

Three batches of SmBCO–Ag single grains of diameter 20, 25 and 31 mm were fabricated using the new buffering technique. T_m and holding time for each heating process was maintained at 1071 °C and 20 min, respectively. The complete heating profile is shown in figure 3(a), with the cooling rate over the temperature range between 1024 and 1004 °C reducing from 0.5 to 0.2 °C hr⁻¹ for increasing sample diameter from 20 to 31 mm. The fully grown single grains were annealed in 1% O₂ in Ar at 850 °C for 3 days [9] before being oxygenated in flowing O₂ (99.9%) at 380 °C for 10 days. The top surface of every sample was polished flat for trapped field measurements. Each sample was field-cooled to 77 K using liquid nitrogen in a field of 1.3 T applied perpendicular to its top surface. The applied field was then removed and the

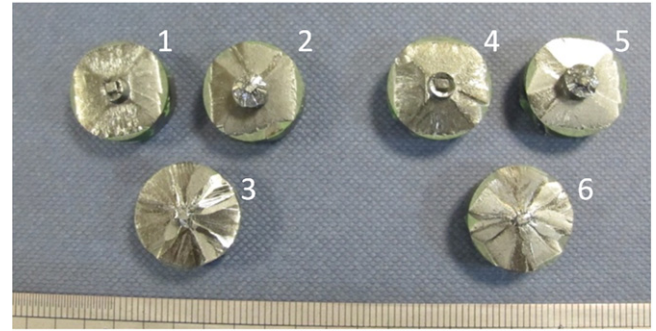


Figure 4. Photographs of the top surfaces of the samples grown using the different seed arrangements shown in figure 2.

trapped field on the top surface of each sample measured using a rotating array of 20 Hall probes. The distance between the sample surface and the Hall probes was estimated to be 0.7 mm.

In order to estimate local T_c and J_c , a randomly chosen SmBCO–Ag single grain of diameter 20 mm was cut into small specimens as illustrated schematically in figure 3(b) (taken from [10]). A MPMS XL SQUID magnetometer was used to measure T_c and M – H loops for increasing and decreasing field cycles for each specimen. The extended Bean model [11] was used to estimate J_c for the orthorhombic samples using the formula $J_c = 20m/(abc)/(a(1-a/3b))$ (A cm⁻²), where m (emu) represents the difference in magnetic moment observed in the M – H loops for increasing and decreasing field cycles with the applied field perpendicular to the ab -plane. Here a , b and c are the dimensions of the specimens along the principal crystallographic axes (where $a < b$), and are measured in centimetres.

3. Results and discussion

3.1. Reliability of buffering technique for fabricating SmBCO–Ag single grains

Figure 4 shows that the results obtained from the TSMG processing of the two sets of three samples are the same. In each case, the buffer layers of composition of 75 wt% Sm-123 + 25 wt% Sm-211 survive the melt process for a T_m of

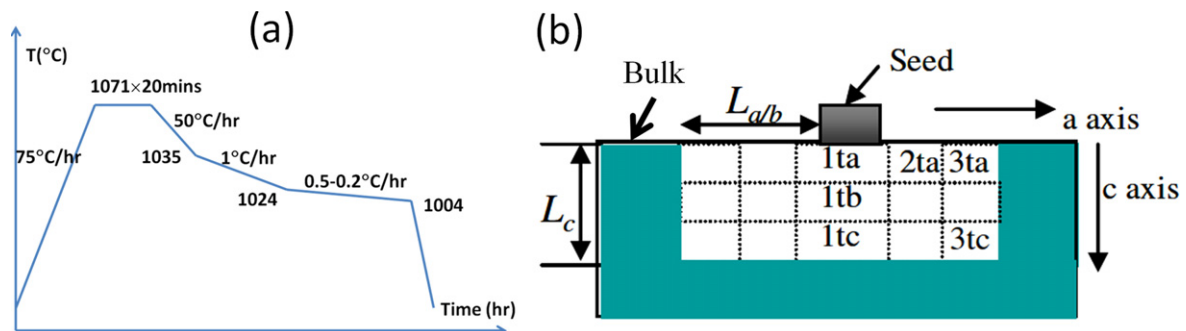


Figure 3. (a) Heating profiles used for batch-processing SmCO–Ag single grains of different sizes from 20 to 31 mm in diameter. The cooling rate in the growth window reduces as sample diameter increases. (b) Schematic illustration of the location of the sub-specimens for characterization of the superconducting properties in the parent single grain [10].

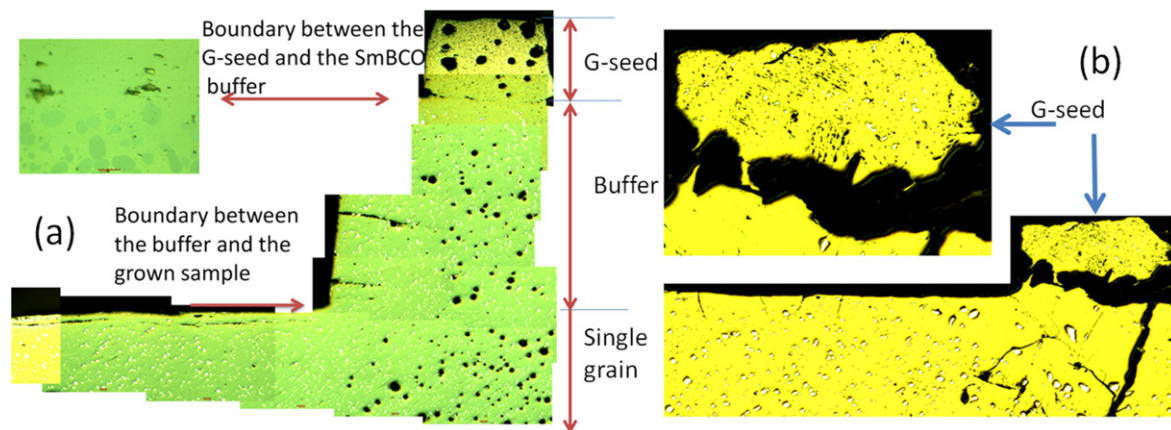


Figure 5. (a) Microstructure of the cross-section of sample 1 (shown in figure 4) along the crystallographic c direction (i.e. pellet thickness) and through the position of the seed (b) microstructure of the cross-section of sample 2 (shown in figure 1) along the crystallographic c direction (i.e. through the pellet thickness) and through the position of the seed.

1071 °C and a holding time of 20 min. The pellets seeded using the Sm-211 buffer and those seeded directly, however, all failed under the same processing conditions.

Figure 5(a) shows the microstructure of the sample cross-section along the crystallographic c direction (i.e. pellet thickness) and through the position of the seed of sample 1 (labelled in figure 4). Figure 5(b) shows the microstructure of the cross-section of sample 2 (labelled in figure 1) in the vicinity of the failed seed. It can be seen from the inset of figure 5(b) that silver (white in contrast in the photograph) has diffused into the seed. Furthermore, the seed failed to retain its shape and even lost partial contact with the seeded pellet during the melt process. It is clear, therefore, that this seed is obviously not functional. It can be seen from figure 5(a) that Ag (white in contrast in the photograph) is distributed throughout the sample and present mainly within the pores left by the residual porosity from the melt process. It can be seen further that Ag permeates into the buffer layer during melt processing, but terminates before it reaches the interface between the seed and the buffer. The inset photograph was taken at a magnification of 1000 $\times$, from which it can be seen more clearly that no Ag is present within the generic seed microstructure. As a result, the melting temperature of the seed and lattice structure were not altered during melt processing, enabling the generic seed to function well under the melt growth conditions, even though the seed exhibits a relatively large amount of porosity.

A buffer of composition of 75 wt% Sm-123 + 25 wt% Sm-211 is now used routinely by the Cambridge Bulk Superconductivity Group to process SmBCO–Ag using TSMG based on the results of this study, which has enabled large SmBCO–Ag (10 wt% Ag₂O) single grains to be fabricated reliably. Figures 6(a) and (b) show samples of diameter 25 and 31 mm respectively fabricated using the buffer layer technique. In total, about 50 single grain samples have been seeded recently using the buffer layer technique with a success rate of 100%. (Significantly, this technique can also be used to grow GdBCO–Ag single grains, which are equally easy to seed when a buffer layer is employed.)

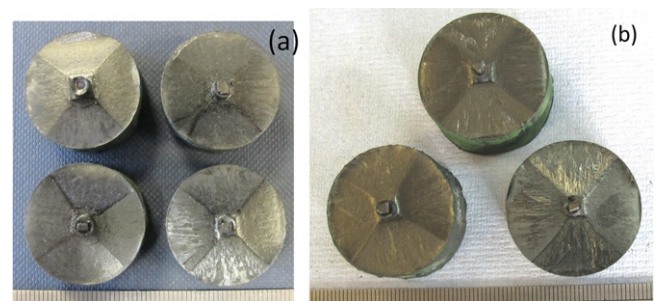


Figure 6. SmBCO–Ag single grain samples of diameter (a) 25 and (b) 31 mm fabricated using a buffer layer.

3.2. Superconducting properties SmBCO–Ag single grains fabricated using the buffering technique

Figures 7(a) and (b) show the measured critical temperatures, T_c , and estimated values of J_c calculated using the extended Bean model. It can be seen that most of the T_c curves exhibit a high onset T_c (92–93 K) and a sharp superconducting transition, except for the data for specimens 2ta and 3ta. The J_c curves for specimen positions other than 2ta and 3ta exhibit a characteristic shape, including a relatively high $J_c(0)$ and a peak effect at about 1.5 T. The low J_c for specimens 2ta and 3ta is expected given the relatively low T_c and broad transition width ΔT_c of these samples (the measurement temperature of the $M-H$ loop is carried out at 77 K and is closer to the $T_c - \Delta T_c$ of these samples, which reduces J_c). Takebayashi [12] pointed out that ‘large Sm–Ba–Cu–O samples processed in air, T_c could be enhanced by using the Ba-rich initial composition and the low temperature growth. However, the spatial variation in the superconducting properties of these samples was so large that multiple peaks were observed in the trapped field distribution’. The observed lower T_c for specimens 2ta and 3ta suggests that Sm/Ba substitution is not suppressed sufficiently for these two positions in the parent single grain, perhaps due to the close proximity of these two locations to the seed, which are not in the low temperature growth region. The differences in peak value of J_c and peak position of J_c of each sample are associated with the size and

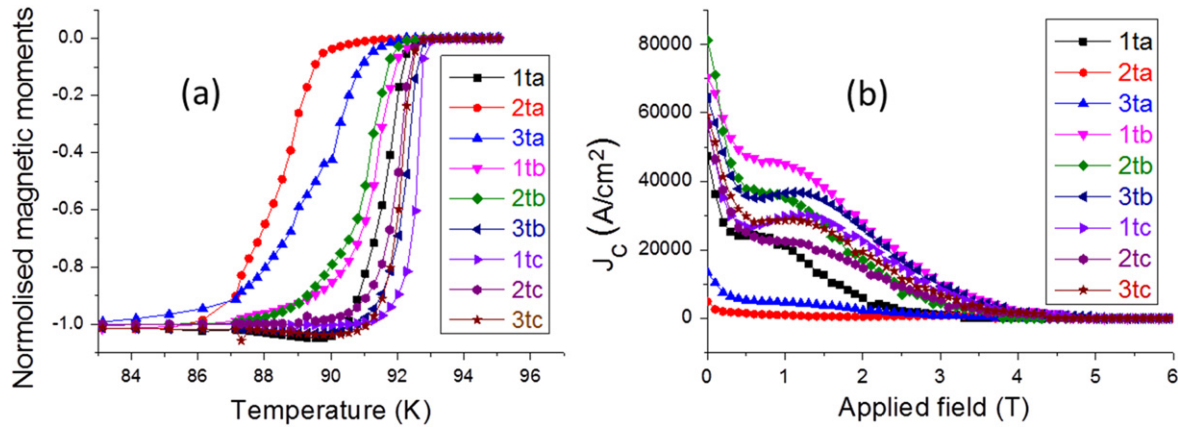


Figure 7. The superconducting properties of nine specimens cut from different positions in a SmBCO–Ag single grain fabricated using a buffer layer (a) T_c measurements (b) J_c estimations.

number of $\text{Sm}_{1-x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_{7-\delta}$ clusters present in the small specimen used for J_c measurement (finely distributed LRE-rich $\text{LRE}_{1-x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_{7-\delta}$, where LRE=Nd, Sm and Gd, clusters of size 10–50 nm can act as field-induced pinning centres [13]). The size of $\text{LRE}_{1-x}\text{Ba}_{2-x}\text{Cu}_3\text{O}_{7-\delta}$ clusters would be associated with their solubility and related further the heating profiles and post-annealing process, which will be the subject of further research.

In addition to an aid to seeding, another motivation for using the buffers is to enhance the superconducting properties of the specimen under the seed (i.e. at position 1ta) where both T_c and J_c are typically low due to contamination from the seed and reduced flux pinning in this region [14, 15]. It can be seen that the blue curve in figure 7(a) has an onset T_c of 92.5 K and exhibits a rather sharp transition, although it is not the best T_c curve measured. The J_c curve of specimen 1ta (light purple) is also reasonable with a $J_c(0)$ of $5.0 \times 10^4 \text{ A cm}^{-2}$ and a peak effect at 1 T. The observed values of T_c and J_c for the specimen located immediately below the seed indicate that buffers used in this research not only prevent Ag infiltrating the seed to enhance the reliability of the seeding process, but also limit the harmful effects of the elements Nd and Mg diffusing from the seed to the single grain. A direct consequence of this is that the superconducting properties of the SmBCO–Ag single grain specimen under the position of the seed are increased to the level reported in previous studies [14, 15]. The differences in T_c and J_c of the nine specimens suggest that Sm/Ba substitution is not totally suppressed in the research. However, the buffer technique clearly has no negative effects on the superconducting properties of the seeded samples.

Figure 8 shows photographs of the top surfaces of five polished SmBCO–Ag single grains. The trapped fields of these samples measured at 77 K are indicated in the figure. These values are higher than the average value (0.5 T) observed typically for YBCO single grains of these dimensions, but lower than the average value (0.7 T) observed for GdBCO–Ag single grains. Although the values of trapped field obtained in this research are the highest reported in the SmBCO–Ag system for samples this diameter, there is still

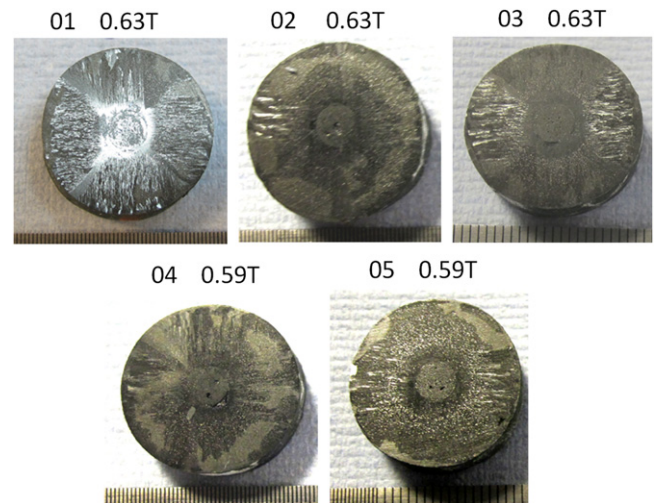


Figure 8. Photographs of the top surface of five SmBCO–Ag single grains of diameter 20 mm. The trapped fields are as indicated.

potential to improve its superconducting properties by further suppression of Sm/Ba substitution effects.

Figure 9 shows the average trapped field profiles of the SmBCO–Ag single grains of diameter 25 and 31 mm, shown in figures 6(a) and (b). Average values 0.60 T for the 25 mm diameter samples and 0.65 T for 31 mm diameter samples are obtained. The variations in the average value of trapped field across all the samples of a given diameter are shown in the figure. Although a single grain of diameter 31 mm is the largest SmBCO–Ag single grain reported to date, the observed magnitude of trapped field for these larger samples is significantly lower than is expected for a fully superconducting sample of this size. The reason for the observed low values of trapped field is the severe effect on T_c of Sm/Ba substitution that occurs when the melt processing is performed in an air atmosphere. However, the necessary annealing process in 1% O_2 in Ar and subsequent oxygenation have not yet been optimized for samples larger than 20 mm, and it is anticipated that increased trapped fields will be produced when this optimisation has been performed.

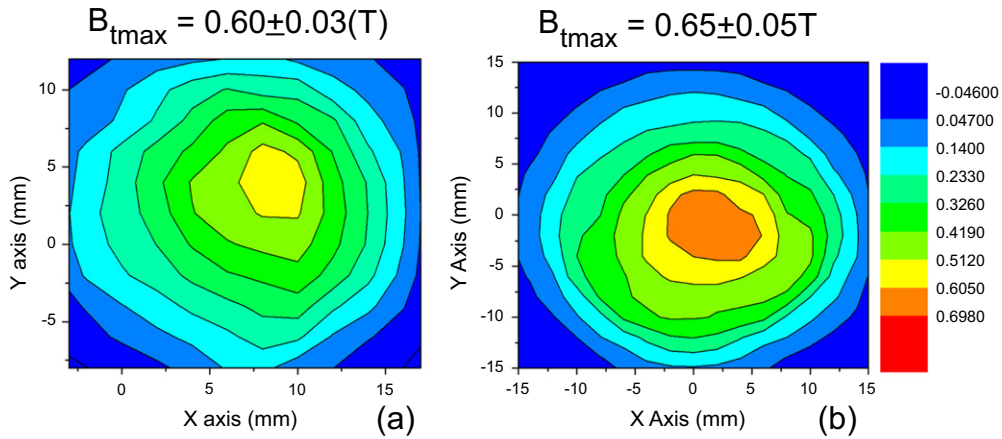


Figure 9. Average trapped fields of the SmBCO–Ag single grains of diameter (a) 25 and (b) 31 mm.

3.3. The function of the buffer

Several attempts to use a buffer layer have been made previously in the TSMG process [14, 15] with the specific aim of preventing contamination of the precursor pellet with Nd, Mg or Sm from the seed positioned on the upper surface of the buffer, which are evidenced by the high T_c and narrow ΔT_c at the position under the buffer compared to the position directly under the NdBCO deposited MgO film [14] and SmBCO seed [15]. However, the buffer layer technique developed here focuses on inhibiting the diffusion of Ag into the single crystal seed from the bulk sample during melt processing (which can be seen from figure 5), which is an extension of these earlier studies. Measurements on specimens under the buffer at position 1ta exhibit a higher and sharper T_c and high J_c , which indicates that the material at this position is not contaminated by Nd and Mg from the G-seed. As a result, the buffers used in this study function to inhibit diffusion of impurities (Nd, Mg in one direction and Ag in the other) from both the top and bottom of the buffer, so that the seed can survive and function consistently under relatively severe processing conditions to enable reliable batch processing of SmBCO–Ag.

Zhou *et al* suggested that buffers can reduce the lattice mismatch between the seed and the precursor pellet [16, 17], and this is supported by the results of the current study. It is apparent that the lattice mismatch between the seed and the grown single grain can be very small if only the parameters of the respective single cells are compared (in the present case, the difference in lattice parameters a , b , and c between the generic seed and grown single grain SmBCO–Ag is of the order of 0.1 Å). However, the difference in length caused by lattice mismatch in the seeding region (typically $\sim 2 \times 2 \text{ mm}^2$) can increase to several tenths of one mm in one dimension, which can cause failure of the seeding process. When a buffer is used, this accumulated lattice mismatch can be overcome by expanding the buffer layer volume into the processing atmosphere (i.e. obstacle free and air in this investigation). In this case, the grown buffer functions subsequently as a more effective seed in terms of impurity content and lattice compatibility, given that its composition is very close to that of

the target single grain. Figure 10 shows two, randomly chosen buffer layers and their seeds used to melt-process the single grain samples described in this study, from which it can be seen clearly that each buffer has, itself, grown into a single grain. Facet lines are clearly visible on both the top and side surfaces of these samples, which is indicative generally of successful TSMG. These fully-grown single grains with buffers are structurally better than those grown directly with a generic seed in terms of lattice compatibility with the SmBCO–Ag pellet. As a result, SmBCO–Ag single grains can be grown easily and reliably using a buffer layer.

Experience based on the growth of over a hundred GdBCO–Ag and SmBCO–Ag single grain samples, with and without buffer layers, suggests that the buffer layer technique also aids the seeding process itself. The buffer technique, which is well known in the field of crystal growth [18, 19], effectively stabilizes the seed and filters impurities from the seeding and growth process [18, 19]. The buffers used in this study functioned additionally as seeds with fewer defects, which suggests the buffering technique is potentially applicable to other (RE)BCO systems for increasing reliability of the TSMG process. It may be concluded, therefore, that a buffer layer of composition 75 wt% Sm-123 + 25 wt% Sm-211 has been identified to stabilize the decomposition temperature of the generic Mg–NdBCO seed and acts as an effective barrier to impurities during processing, such as Nd, Mg, Ag and CeO_2 in the present case, which are potentially harmful to the seeding process. Further research, however, is needed to investigate and understand the mechanism in more detail.

4. Conclusions

A buffer layer of composition of 75 wt% Sm-123 + 25 wt% Sm-211, diameter 5 mm and weight 0.3 g has been identified as an effective aid to the seeding process using a generic NdBCO(MgO) single crystal seed. A success rate of 100% of the seeding process has been achieved using the buffer layer technique, which is an essential requirement for fully growing single grains on a large scale. Large, SmBCO–Ag single

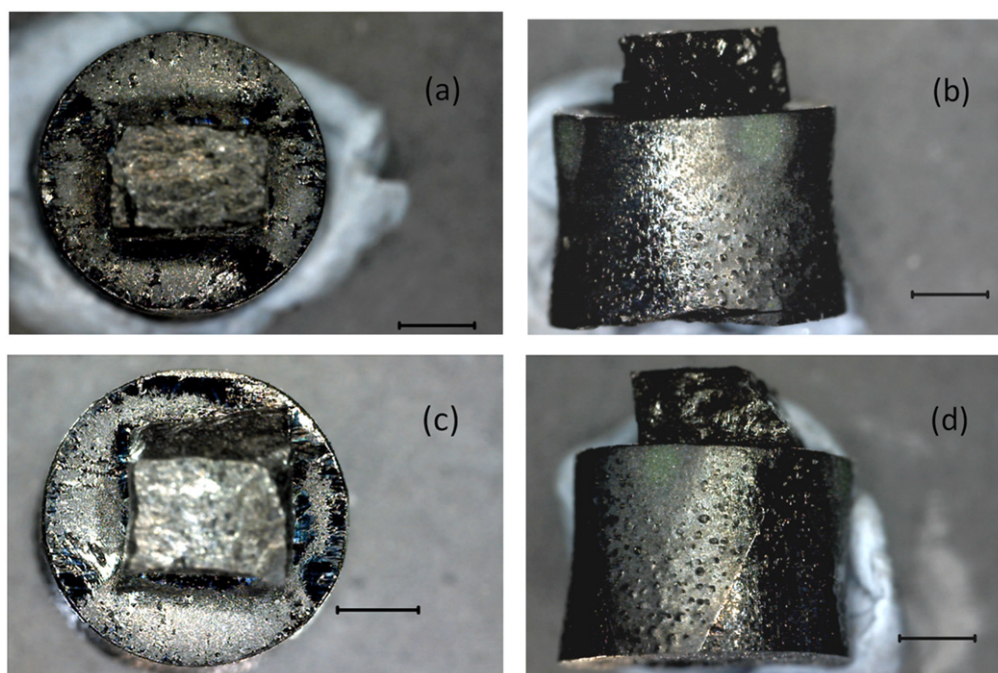


Figure 10. Photographs of buffer layers and their seeds after melt processing (the scale bar represents 1 mm).

grains of diameter up to 31 mm have been batch-fabricated successfully using this technique. Analysis of the sample microstructure and the features of the buffer layer and seed after melt processing suggests that the presence of the buffer layer stabilizes the seed and presents an effective physical barrier to contamination of the single crystal seed by impurity elements, and by Ag, in particular, in the present study. The superconducting properties of the SmBCO–Ag single grains are not affected by the buffer-seeding process. Record trapped fields have obtained in the SmBCO–Ag system using the buffer layer, although further research is required to suppress Sm/Ba substitution in the TSMG process for these materials fabricated in an air atmosphere.

Acknowledgments

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