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A novel, two-step top seeded infiltration and growth process for the fabrication of single grain, bulk (RE)BCO superconductors

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Abstract

A fundamental requirement of the fabrication of high performing, (RE)–Ba–Cu–O bulk superconductors is achieving a single grain microstructure that exhibits good flux pinning properties. The top seeded melt growth (TSMG) process is a well-established technique for the fabrication of single grain (RE)BCO bulk samples and is now applied routinely by a number of research groups around the world. The introduction of a buffer layer to the TSMG process has been demonstrated recently to improve significantly the general reliability of the process. However, a number of growth-related defects, such as porosity and the formation of micro-cracks, remain inherent to the TSMG process, and are proving difficult to eliminate by varying the melt process parameters. The seeded infiltration and growth (SIG) process has been shown to yield single grain samples that exhibit significantly improved microstructures compared to the TSMG technique. Unfortunately, however, SIG leads to other processing challenges, such as the reliability of fabrication, optimisation of RE_2BaCuO_5 (RE-211) inclusions (size and content) in the sample microstructure, practical oxygenation of as processed samples and, hence, optimisation of the superconducting properties of the bulk single grain. In the present paper, we report the development of a near-net shaping technique based on a novel two-step, buffer-aided top seeded infiltration and growth (BA-TSIG) process, which has been demonstrated to improve greatly the reliability of the single grain growth process and has been used to fabricate successfully bulk, single grain (RE)BCO superconductors with improved microstructures and superconducting properties. A trapped field of ~0.84 T and a zero field current density of 60 kA cm^{-2} have been measured at 77 K in a bulk, YBCO single grain sample of diameter 25 mm processed by this two-step BA-TSIG technique. To the best of our knowledge, this value of trapped field is the highest value ever reported for a sample fabricated by an infiltration and growth process. In this study we report the successful fabrication of 14 YBCO samples, with diameters of up to 32 mm, by this novel technique with a success rate of greater than 92%.

Keywords: infiltration growth, melt growth, buffer, YBCO bulk, single grains, trapped field, superconducting properties

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

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1. Introduction

Bulk, single grain RE–Ba–Cu–O ((RE)BCO, where RE is a rare-earth element) superconductors have significant potential for a variety of high field engineering applications at cryogenic temperatures, including trapped field magnets, friction

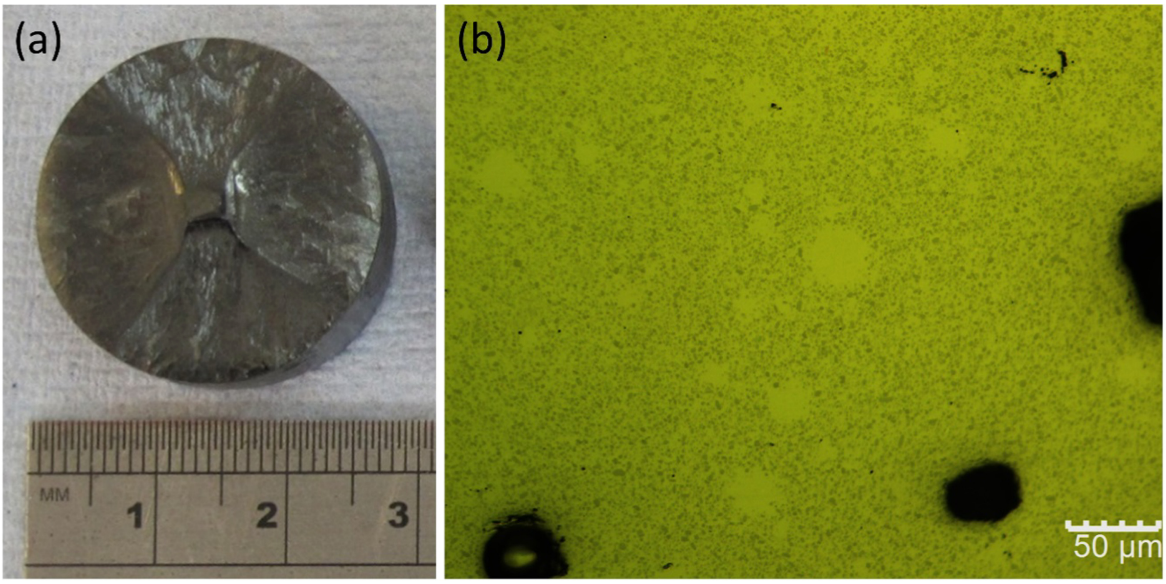

Figure 1. (a) Top view of a single grain YBCO sample fabricated by a conventional top seeded melt growth (TSMG) process. (b) Shows the presence of pores and regions free of Y-211 phase inclusions within the sample microstructure.

free, self-stabilising bearings for energy storage flywheels and magnetic separation devices [1, 2]. As a result, several processing techniques have been developed over the past 25 years to fabricate bulk (RE)BCO superconductors in single grain form. Of these, the so-called top seeded melt growth (TSMG) process is the most established and widely used technique for fabricating high quality single grain bulk samples [3–10]. The field trapping ability of (RE)BCO single grains processed by TSMG is determined largely by the product of the critical current density, J_c, and the extent, r, over which this current can flow within the bulk microstructure. The latter, in turn, depends on the size of the single grain. A record trapped field of 17.6 T has been observed recently in an arrangement of two GdBCO/Ag single grain bulk samples at 26 K, which underlines the potential field-generating performance of these technologically important materials [6].

Although the TSMG technique yields high quality (RE) BCO bulk single grain samples that exhibit superior superconducting properties, there are certain intrinsic problems [11, 13–16] associated with the TSMG fabrication process itself. These include sample shrinkage during processing and the presence of macro-defects, such as extensive porosity and regions free from non-superconducting $(RE)_2BaCuO_5$ (RE-211) phase inclusions, which contribute effectively to flux pinning, within the bulk TSMG microstructure, as illustrated in figure 1. The formation of these defects is unavoidable due to the outflow of the liquid phase during the heat treatment, the open-porosity under which the green pellets are made and the incongruent melting of $(RE)Ba_2Cu_3O_{7-\delta}$ (RE-123) phase, which results in the release of oxygen gas at the processing temperature [15, 16]. The top seeded infiltration and growth (TSIG) process overcomes many of these limitations of the TSMG technique [12–22].

The conventional TSIG process involves placing a RE-211 preform capped with a seed crystal in contact with a liquid phase reservoir and then subjecting the arrangement to an appropriate thermal treatment. The Cu-rich liquid phase (comprising $BaCuO_2$ and CuO) originating from the liquid phase reservoir infiltrates into the RE-211 preform during the TSIG process, and reacts subsequently with the RE-211 phase to form the superconducting $(RE)Ba_2Cu_3O_{7-\delta}$ (RE-123) phase. At this stage, heterogeneous nucleation is initiated by the seed crystal, which promotes subsequently the growth of RE-123. The absence of melting of the RE-211 preform helps obviate problems of shrinkage and porosity. Although the TSIG process is interesting microstructurally, the success rate of single grain growth is typically marginal and the corresponding superconducting properties achieved are generally inferior in comparison to samples processed using TSMG, which limits the effectiveness of this process. The primary reason for the poor success rate of TSIG is associated with the complexity of the single grain growth process. Recent work [23] has suggested that the characteristic presence of a large volume fraction of the RE-211 phase in fully processed TSIG samples affects critically the superconducting properties, and hence performance, of the single grain bulk samples.

This paper describes the development of a novel, two-step buffer-aided top seeded infiltration and growth (two-step BA-TSIG) process for the fabrication of high quality single grain bulk YBCO superconductors. This approach provides additional scope for fine-tuning the properties of the non-superconducting phase, which results directly in improved superconducting and microstructural properties. This new fabrication process involves two critical and independent steps: one promotes the infiltration process and the other aids heterogeneous nucleation and subsequent grain growth. In addition, this technique enables potentially the fabrication of near-net shaped bulk products in the form of a large single

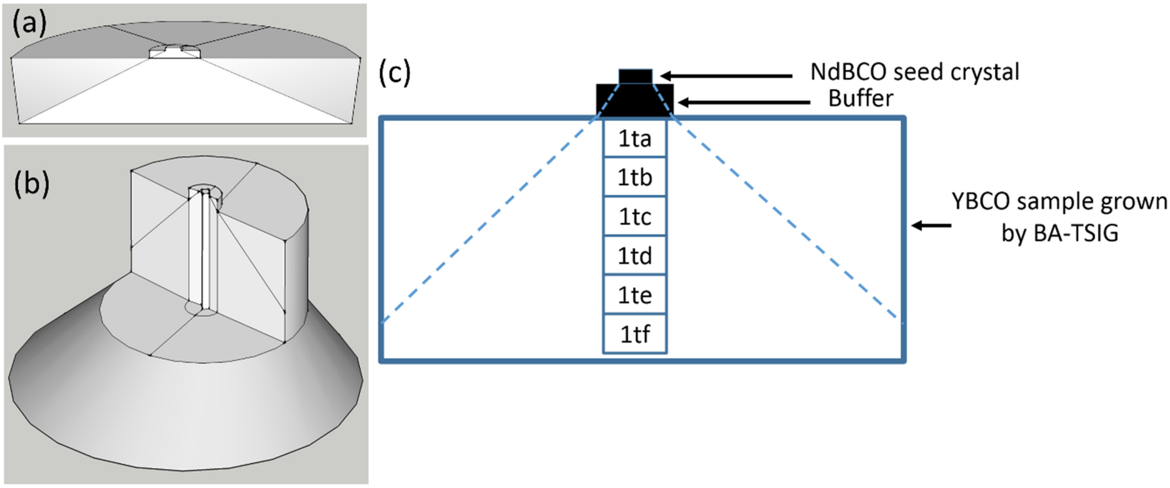

Figure 2. Schematic illustration of the preparation of sub-specimens for (a) the BA-TSMG and (b) the BA-TSIG processed YBCO samples. (c) Shows schematically the position of the specimens extracted from the bulk samples for measuring the superconducting properties of each single grain.

grain. A number of YBCO samples with diameters up to 32 mm have been fabricated successfully using this technique and their properties compared with those of control YBCO samples fabricated using existing BA-TSMG and BA-TSIG processes. Detailed studies of the microstructural and the superconducting properties of the samples fabricated by the two-step BA-TSIG process are presented.

2. Experimental details

Commercial powders of Y-123, Y-211, $Ba_3Cu_5O_8$ (each of 99.9% purity, Toshima), Yb_2O_3 (99.9%, Alfa Aesar), CeO_2 (99%, Sigma Aldrich) and BaO_2 (95%, Sigma Aldrich) were used to fabricate the single grain YBCO samples. A turbula mixer (Willy A. Bachofen model T2F) was employed to ensure that the constituent powders were mixed homogeneously prior to melt processing. The following section describes details of sample fabrication, followed by a comparison of the performance of three YBCO samples (A, B and C) fabricated using the BA-TSMG, BA-TSIG and the newly developed two-step BA-TSIG processing techniques, respectively.

The sample assemblies were subjected to a thermal profile that included initial heating to 1055 °C, holding the temperature for about 1 h, rapid cooling to 1010 °C and then a slow cooling at 0.7 °C–0.5 °C h^{-1} to 980 °C. All the fully grown YBCO samples were annealed in flowing oxygen in the temperature range 450 °C–430 °C for between 150 and 200 h. Each sample was field cooled to 77 K in the presence of an applied magnetic field of 1.2 T, with $\boldsymbol{B}$ applied parallel to the crystallographic c-axis of the sample. The applied field was then removed and the trapped magnetic flux density at the top surface of each sample measured initially by a hand held Hall probe, and then by an automatic, scanning Hall probe system comprising of an array of 19, uniformly spaced probes. The air gap between the sample surface and the Hall probe array in the later measurement was approximately between 0.8 and 1 mm. Sections of the fully processed, oxygenated YBCO single grains were prepared using a diamond saw (Minitom, Struers) and sub-specimens were extracted from the parent grain at the locations shown schematically in figure 2. A SQUID magnetometer was used to measure the magnetic hysteresis M-H loops at 77 K of the sub-specimens for all three YBCO samples by applying a field of up to 6 T. Critical current densities were estimated subsequently from the width of the M-H loops using the extended Bean critical state model [24].

Samples A, B and C were sliced vertically and then polished down to 1 μm using SiC paper and subsequently with diamond paste using a mechanical polisher (Knuth Rotor 2 and Struers DAP-7) in a two-step process. The microstructures of each polished sample were studied using an optical microscope (Nikon, ME600) equipped with a polariser.

3. Results and discussion

Recent work [16, 23, 25–28] has shown that the use of a buffer pellet between the precursor pellet and the seed crystal prior to melt processing prevents any melting of the seed. The presence of the buffer also inhibits diffusion of components of the seed crystal, e.g. Nd, Sm and impurities, into the bulk, single grain microstructure. The buffer pellet, therefore, absorbs minor distortions present in the seed crystal and enables the main bulk sample to grow into a single grain. A buffer pellet capped with a seed crystal acts effectively as a large seed and works in a similar way to a hot seed, overcoming any potentially serious lattice mismatch effects between the seed and the target single grain Y-123 phase.

Table 1. Dimensions and weights of the three YBCO samples A, B and C fabricated employing BA-TSMG, BA-TSIG and the newly developed two-step BA-TSIG technique.

		Dimensions of the as-made YBCO/Y-211 precursor pellet			Dimensions of the YBCO sample, after heat treatment				
Sample code	Fabrication technique	Height (mm)	Diameter (mm)	Weight (g)	Height (mm)	Diameter (mm)	Weight (g)	Change in diameter (%)	Change in weight (%)
Sample-A	BA-TSMG	18.1	31	44	14.5	25.3	41.8	−18.4%	−5%
Sample-B	BA-TSIG	11.3	25	20	12.0	25.4	37.2	+1.6%	+86%
Sample-C	Two-step BA-TSIG	11.3	25	20	12.2	25.6	38.0	+2.4%	+90%

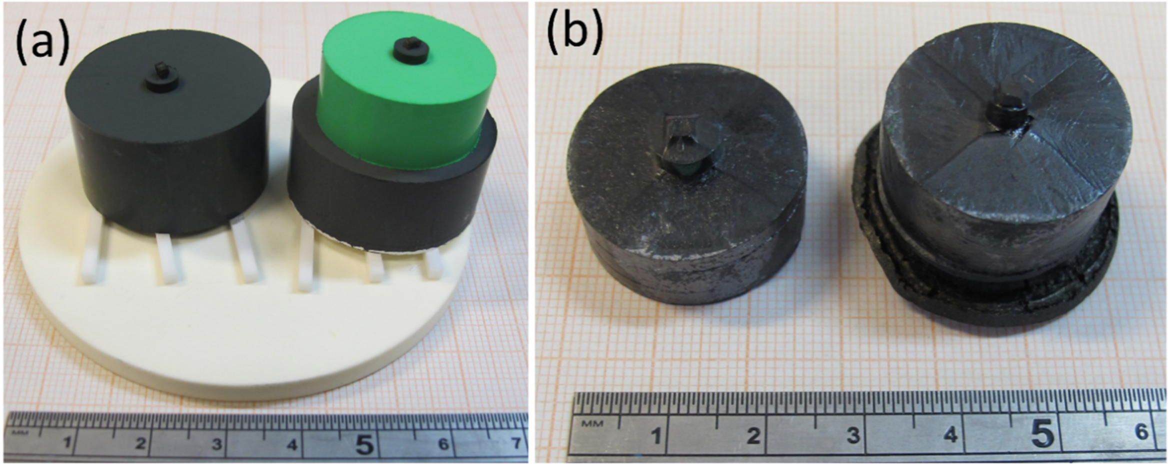

Figure 3. Photographs of the (a) initial A and B sample assemblies (i.e. prior to heat treatment) and (b) the corresponding as-processed YBCO samples. Samples A and B were processed by the BA-TSMG and BA-TSIG techniques, respectively.

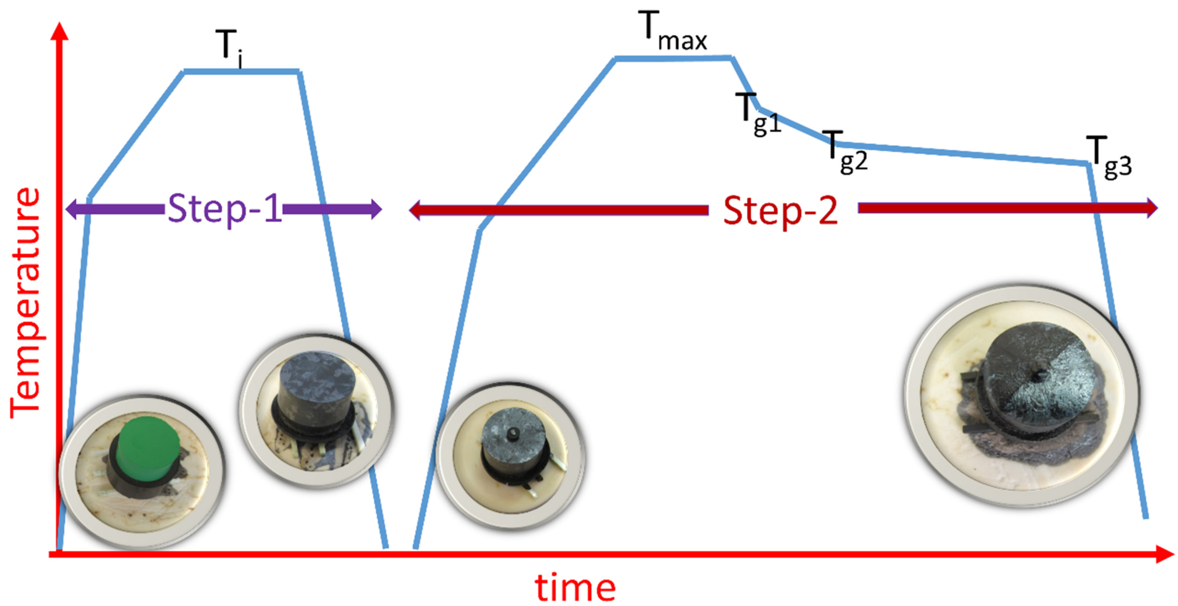

Figure 4. Schematic diagram of the individual steps in the two-step BA-TSIG process. The sample assembly at different stages of processing is illustrated in the figure.

3.1. Fabrication of samples A and B

A single grain YBCO sample was prepared using a conventional buffer-aided TSMG technique. The composition of the precursor pellet for this sample comprised 75 wt% Y-123 + 25 wt% Y-211 + 0.5 wt% CeO_2. A buffer pellet of 5 mm diameter capped with Nd-123 cleaved along (00*l*) was used as seed crystal. Further details of the sample preparation by the BA-TSMG technique can be found elsewhere [27].

The composition of the preform pellet for the preparation of samples B and C was Y-211 + 1 wt% CeO_2, with, based on the results of recent work [16], a Yb-based liquid phase liquid phase reservoir. This liquid phase powder was prepared by mixing the constituent Yb_2O_3, $Ba_3Cu_5O_8$ and BaO_2 powders in a molar ratio for Yb_2O_3: CuO: $BaCuO_2$ of 1:6:10. The buffer pellet retained its composition and size throughout the infiltration and growth process. The dimensions and weights of the precursor/preform pellets and the as-processed samples are summarised in table 1.

The assemblies for samples A and B prior to, and after, melt processing are shown in figure 3.

3.2. Fabrication of sample-C by the two-step BA-TSIG process

The two-step BA-TSIG technique comprises two separate processes. The initial step involves infiltrating liquid phase into the Y-211 preform. In the second step, a buffer pellet capped with the seed crystal is placed on the infiltrated bulk preform and then the entire arrangement is subjected to an appropriate heat treatment to achieve single grain growth.

The Y-211 powder (20 g) and the liquid phase powder (32 g) were each pressed uniaxially, into discs of 25 and 32 mm in diameter, respectively. A thin layer of Yb_2O_3 was used both to support the liquid phase reservoir and to prevent

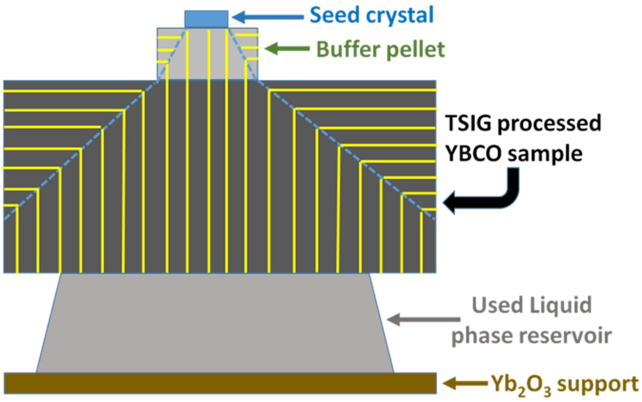

Figure 5. Schematic illustration of the single grain microstructure produced by a buffer pellet capped with a seed crystal to aid the growth of the Y-123 phase in the TSIG process.

the outflow of liquid phase during thermal processing. These pellets were arranged as shown in figure 4 and then subjected to the first step of the two-step BA-TSIG process (the infiltration of the liquid phase into theY-211 preform), as shown in figure 4.

The sample assembly was heated to the infiltration temperature T_i (1050 °C) and held isothermally for sufficient time (1.5 h) to facilitate infiltration of the liquid phase into the Y-211 preform, and cooled rapidly to room temperature immediately after the infiltration step. No seed was employed during this part of the process, which results in the random nucleation of individual YBCO sub-grains within the infiltrated preform microstructure. A buffer pellet composed of Y-123 and Y-211 in a 75:25 weight ratio capped with a cleaved NdBCO seed crystal (along the (00*l*) plane) was placed on the infiltrated sample and subjected subsequently to TSIG processing (i.e. the second step). This involved heating the infiltrated sample assembly to 1055 °C (T_{max}), holding for 0.5 h followed by rapid cooling to 1012 °C (T_{g1}, *growth temperature start*) and then slow cooling at a rate of 0.7 °C–0.5 °C h^{-1} to 980 °C (T_{g3}). Stable heterogeneous nucleation initiates at the seed crystal during this step and growth extends, ultimately, to the edges of the sample, driven by undercooling, to enable the successful fabrication of a single grain YBCO bulk superconductor. The two step process is followed by oxygenation during which the samples were heated in an oxygen atmosphere to a temperature in the range 450 °C–430 °C for between 150 and 200 h.

The use of a buffer pellet aids the single grain growth process significantly, making it much easier and more reliable. The buffer pellet and seed crystal arrangement acts effectively as a single large seed crystal (homo-seed), as illustrated schematically in figure 5.

3.3. Sample microstructure

The superconducting properties of (RE)BCO single grains depends largely on their microstructure, since this controls the pinning of flux vortices and, therefore, J_c. The YBCO samples A, B and C fabricated in this study were examined for Y-211 size, content and distribution. The optical micrographs obtained for each of these samples are shown in figure 6.

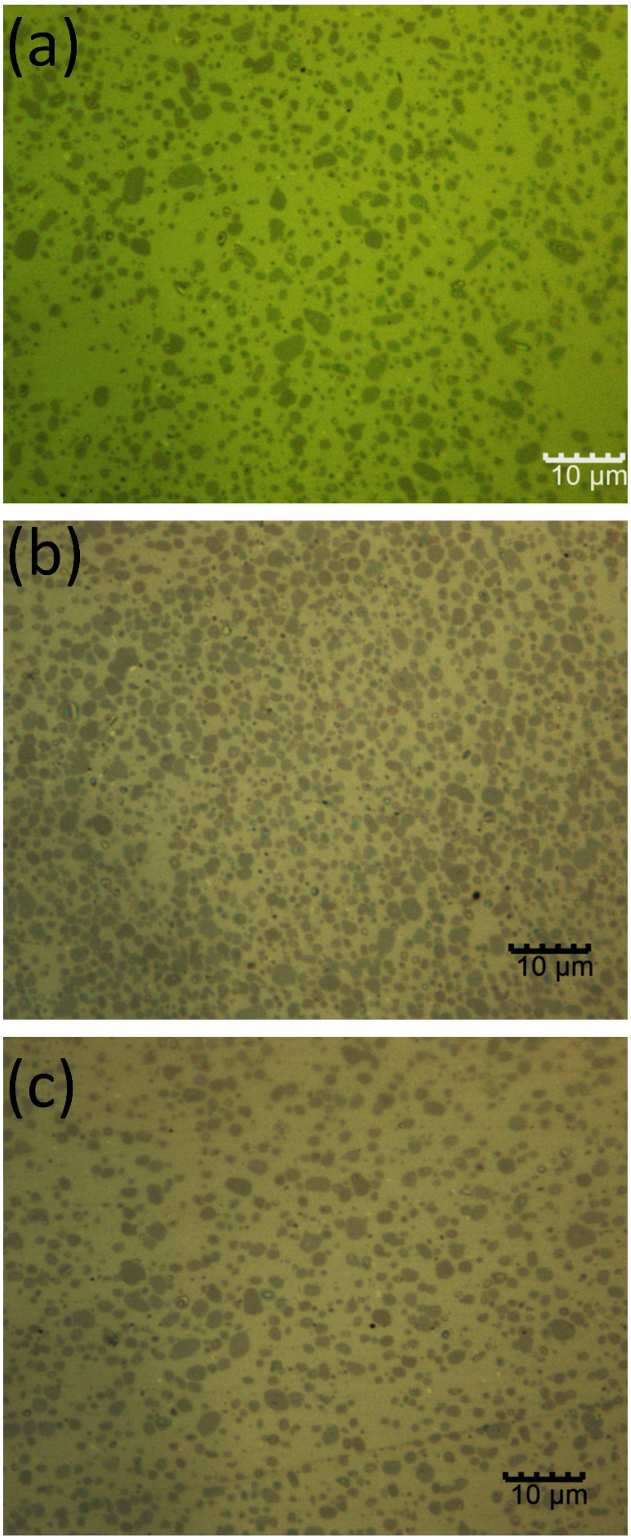

Figure 6. Optical micrographs recorded at the central location (corresponding to position 1tc in figure 2) of the YBCO samples fabricated using (a) BA-TSMG, (b) BA-TSIG and (c) two-step BA-TSIG processing techniques. The size and distribution of Y-211 inclusions in the matrix of Y-123 in each of the samples can be seen clearly.

Table 2. The magnitude of trapped fields measured at the surface of the YBCO samples at 77 K.

Trapped field measured (in Tesla) straight on the surface of the YBCO samples at 77 K (T)		
Sample-A (BA-TSMG)	Sample-B (BA-TSIG)	Sample-C (two-step BA-TSIG)
0.78	0.61	0.84

It can be seen that sample A (prepared by BA-TSMG) contains regions that are free of the RE-211 phase, although this is not the case for the samples processed by the two TSIG-based techniques (samples B and C). Furthermore, the Y-211 particles are acicular (or needle-like) in sample A, whereas they are more spherical in samples B and C, which is consistent with previous reports [29, 30]. It is evident from figures 6(b) and (c) that the Y-211 content is significantly higher in sample B than in sample C. The Y-211 content in the final bulk microstructure in each of these samples was investigated using *Image-J* analysis software, which indicated the presence of Y-211 concentrations of ~25.6%, ~41.2% and ~30.6% in samples A, B and C, respectively. This is a clear indication that the two-step BA-TSIG process enables a better control of Y-211 compared to conventional TSIG, and is therefore desirable for producing improved superconducting properties of the single grain.

In addition, the absence of a seed crystal in the first step of the two-step BA-TSIG process facilitates additional control (in terms of varying and tuning the infiltration temperature and time) for adjusting the Y-211 content, and hence the superconducting properties of the fully processed single grain. The maximum processing temperature in a typical melt process is limited by the melting temperature of the seed crystal employed and, in this context, the two-step BA-TSIG technique overcomes this limitation effectively. The two-step BA-TSIG process looks extremely promising, therefore, as a practical process for the reliable fabrication of large bulk YBCO single grains.

3.4. Trapped field performance

Samples A, B and C were field cooled in an applied field of 1.2 T to measure their trapped field properties at 77 K using a hand held Hall probe and also by employing subsequently a rotating, scanning Hall array. The resulting trapped fields are summarised in table 2.

The trapped field profiles recorded at a height of between 0.8 and 1 mm above the top surface of each sample are shown in figure 7.

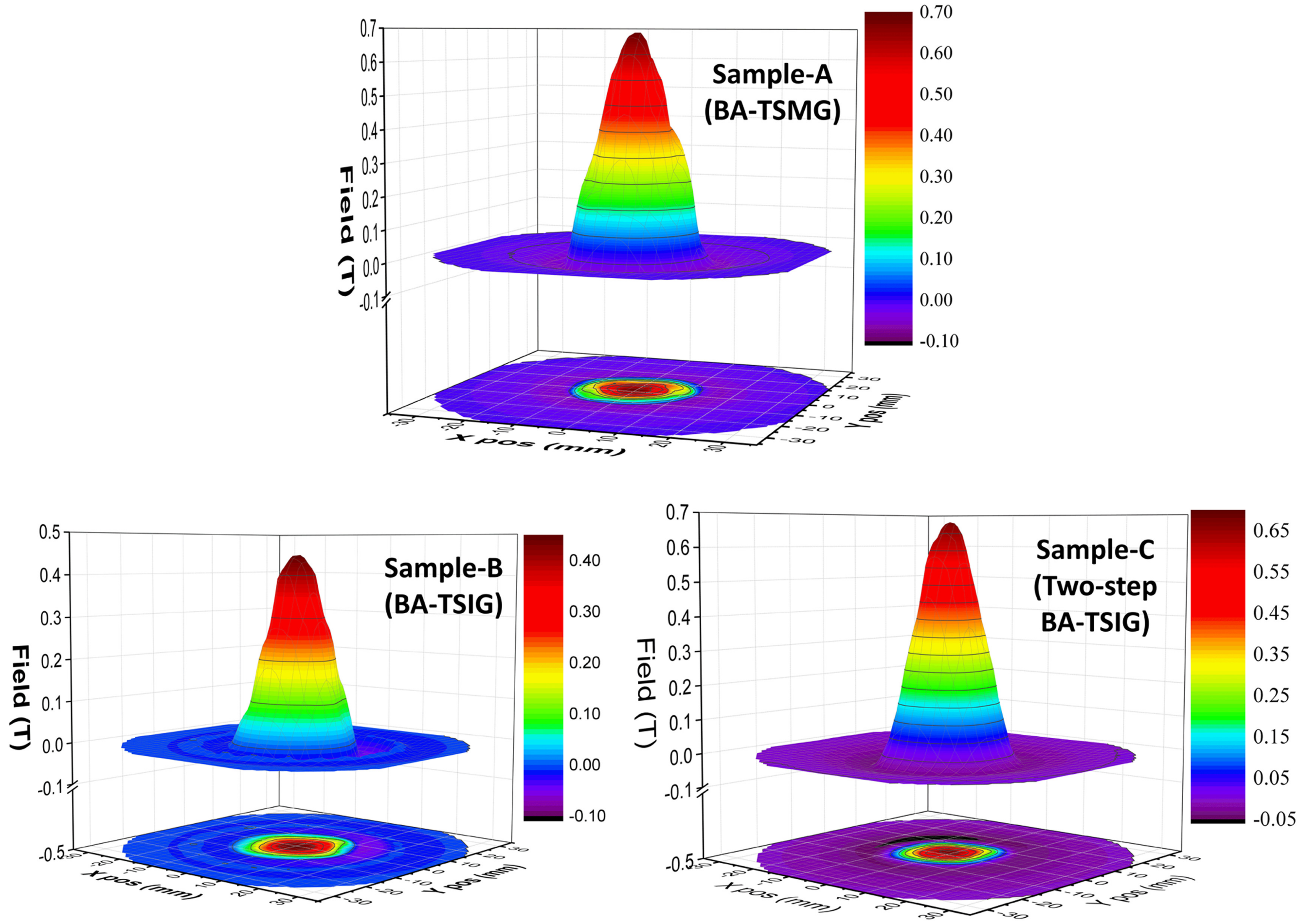

Figure 7. Trapped field profiles obtained for single grain YBCO samples A, B and C. The separation between the Hall sensors and the sample surface in each of these measurements was between 0.8 and 1 mm.

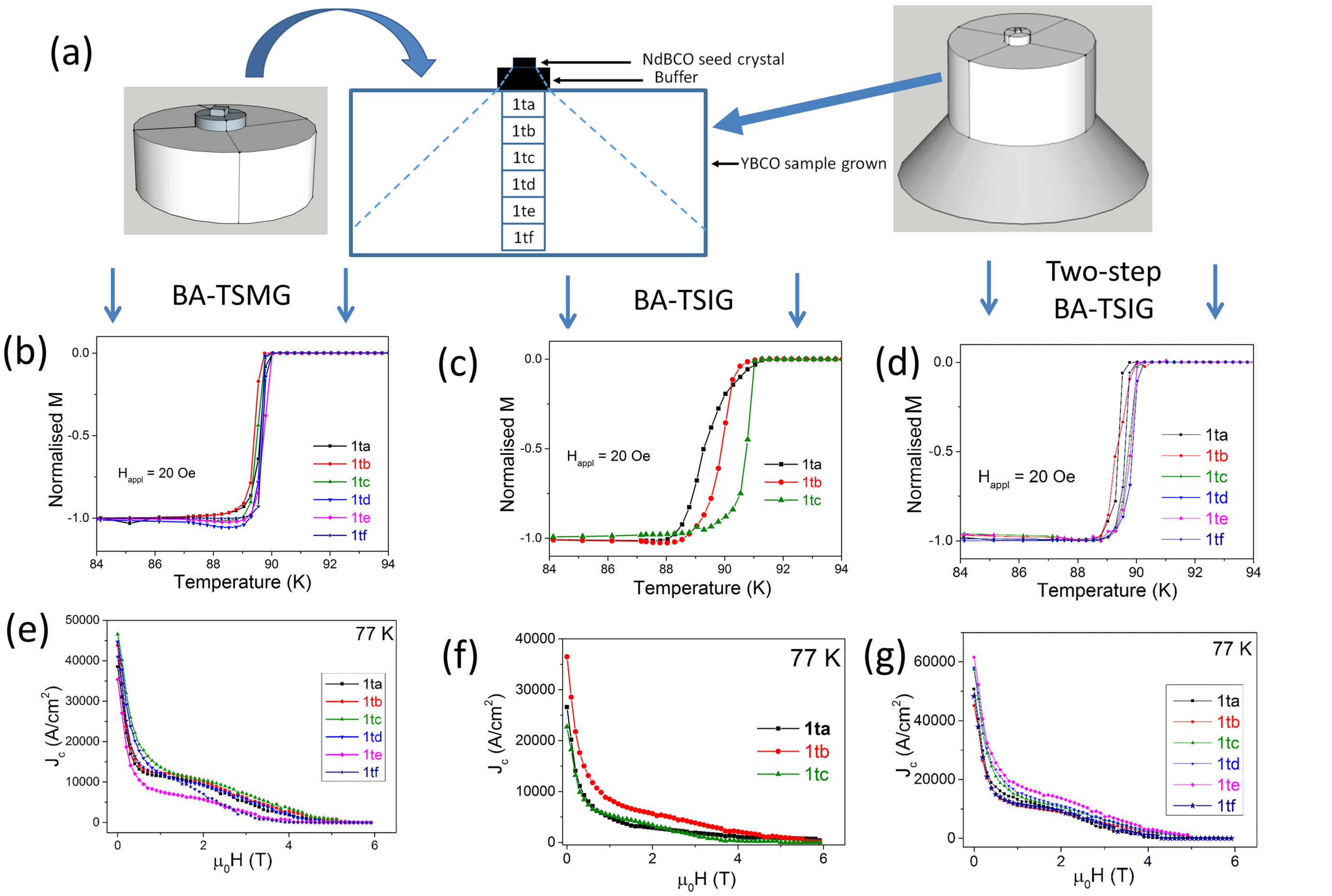

Figure 8. (a) Schematic illustration indicating the positions of the sub-specimens in the parent single grain extracted for magnetisation measurements for reference. (b), (c) and (d) Show the temperature dependences of normalised magnetisation obtained for samples A, B and C, respectively. The field dependences of critical current density J_c obtained for samples A, B and C are shown in (e), (f) and (g), respectively.

3.5. Current density

Magnetisation hysteresis (M-H) loops were measured for the sub-specimens extracted from each of the three YBCO single grain samples. The field dependence of J_c was estimated from the width of the hysteresis loops using the extended Bean model [24]. The results obtained for each of the samples at 77 K are shown in figure 8.

Critical current densities at zero field '$J_c(0)$' were estimated for samples A, B and C to be 46, 36 and 60 kA cm^{-2} at 77 K. It is evident from figure 8 that the superconducting properties are more uniform in samples fabricated by infiltration growth due to a more homogeneous distribution of Y-211 particles in the superconducting Y-123 phase matrix. Sample A exhibited superior performance compared to that of sample B, which is consistent with all samples fabricated in this study. The main reason for the inferior performance of samples fabricated by routine infiltration and growth compared to those processed by TSMG may be attributed to the excess RE-211 content associated in the former.

The two-step BA-TSIG technique, on the other hand, enables fabrication of bulk single grains with an optimum concentration of Y-211 in the final bulk microstructure and addresses simultaneously many of the major limitations of both the TSMG and singe step TSIG processes. Hence, this improved technique has a greater scope for the manufacture of single gains with enhanced superconducting properties. It is evident from figures 6–8 that the two-step BA-TSIG process enables the fabrication of YBCO bulk samples with superior superconducting properties. The reasons for this enhanced performance are twofold: (i) the Y-211 content is successfully tuned and optimised, and (ii) a relatively long oxygenation time of 150–200 h is sufficient for the oxygen to permeate into the dense YBCO single grain microstructure, thereby transforming completely the non-superconducting tetragonal Y-123 phase into the superconducting orthorhombic Y-123 phase [31].

3.6. Reliability and yield

The two-step buffer-aided TSIG process is emerging as far superior processing technique compared to conventional TSIG and is at least comparable to the BA-TSMG process. The single grain growth of samples using this process is also more reliable, due primarily to the use of the buffer layer. Thirteen additional samples have been fabricated

Figure 9. Top view of the YBCO single grain samples fabricated by the novel, two-step buffer-aided TSIG melt process.

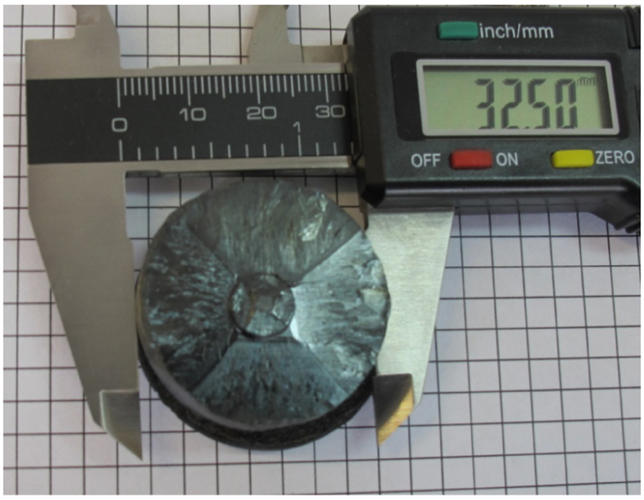

Figure 10. A 32 mm diameter YBCO sample obtained using the two-step BA-TSIG technique.

subsequently via the two-step BA-TSIG technique, as shown in figure 9, which demonstrates the reliability of the process. Only one sample (32 mm in diameter) fabricated by this technique exhibited the presence of a small sub-grain in the single grain microstructure, corresponding to an overall success rate of >92%, which is extremely high for an infiltration growth process. Finally, figure 10 shows a photograph of a YBCO sample of 32 mm diameter fabricated successfully by the two-step BA-TSIG technique, which illustrates the potential of this technique for fabricating large single grain YBCO samples.

4. Conclusions

The seeded infiltration and growth (SIG) technique overcomes some of the fundamental limitations of the TSMG process, such as shrinkage and the formation of RE-211-free regions and micro-cracks. However, the reliability of the SIG growth process and the associated superconducting properties of the resulting single grain samples is inferior compared to the properties of samples fabricated using a conventional TSMG technique. The observed inferior superconducting properties appear to be associated primarily with the large volume fraction of the RE-211phase in the as-processed single grains.

We have developed a successful processing methodology based on a modified infiltration growth technique to allow more scope for tuning and optimising the RE-211 content in the large, bulk YBCO single grains. The two-step BA-TSIG technique involves two key steps to fabricate reliably a large single grain. The first step involves a basic infiltration process, where the liquid phase originating in the liquid phase reservoir infiltrates into the Y-211 preform, and a second step where a seed crystal is placed on the infiltrated bulk to aid the heterogeneous nucleation and subsequent growth of the single grain. This technique provides more scope for controlling the optimum amount of liquid phase entry into the Y-211 preform, which facilitates the adjustment of Y-211 content in the fully processed single grain and therefore optimisation of the superconducting properties. A buffer technique employed to enhance the seeding process has improved significantly the reliability of the single grain growth process.

The samples fabricated by this technique exhibited almost no shrinkage during processing and contained a minimal intrinsic concentration of cracks and pores. An oxygenated YBCO sample of 25 mm in diameter obtained by this novel technique exhibited a trapped field of ~0.84 T at 77 K, which is comparable with that generated by a high quality YBCO single grain sample fabricated by TSMG. Reliability in fabricating YBCO single grain samples by the two-step BA-TSIG technique is good, with an initial success rate >92%, which is encouraging from a production point of view. Finally, several single grains of YBCO of diameter up to 32 mm have been fabricated successfully and reliably as part of this study.

Acknowledgments

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