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A robust seeding technique for the growth of single grain (RE)BCO and (RE)BCO–Ag bulk superconductors

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Abstract

Bulk, single grains of RE–Ba–Cu–O [(RE)BCO] high temperature superconductors have significant potential for a wide range of applications, including trapped field magnets, energy storage flywheels, superconducting mixers and magnetic separators. One of the main challenges in the production of these materials by the so-called top seeded melt growth technique is the reliable seeding of large, single grains, which are required for high field applications. A chemically aggressive liquid phase comprising of $BaCuO_2$ and CuO is generated during the single grain growth process, which comes into direct contact with the seed crystal either instantaneously or via infiltration through a buffer pellet, if employed in the process. This can cause either partial or complete melting of the seed, leading subsequently to growth failure. Here, the underlying mechanisms of seed crystal melting and the role of seed porosity in the single grain growth process are investigated. We identify seed porosity as a key limitation in the reliable and successful fabrication of large grain (RE)BCO bulk superconductors for the first time, and propose the use of Mg-doped NdBCO generic seeds fabricated via the infiltration growth technique to reduce the effects of seed porosity on the melt growth process. Finally, we demonstrate that the use of such seeds leads to better resistance to melting during the single grain growth process, and therefore to a more reliable fabrication technique.

Keywords: bulk superconductor, single grain, generic seed, infiltration and growth, seeding, buffer technique

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

1. Introduction

Bulk, single grain RE–Ba–Cu–O [(RE)BCO, where RE is one of a number of rare earth elements, including Y, Nd, Sm or Gd] have the potential to trap larger magnetic fields than those generated by conventional, iron-based permanent magnetic materials [1–3]. In order to achieve large trapped fields, it is necessary to fabricate bulk (RE)BCO superconductors in the form of large, single grains, since the presence of grain boundaries inhibits the flow of super-current in these materials. The trapped field in a superconductor is known to vary as $J_c{}^* r$, where r is the radius of the super-current loop and J_c is the critical current density of the superconductor [4]. Hence, it is desirable to have large values of both these parameters for optimum field trapping ability. Enhancing J_c through a number of approaches has been attempted by various researchers, but primarily via the addition of non-superconducting phases to the superconducting $(RE)Ba_2Cu_3O_{7-\delta}$ (RE-123) [5] phase composition to obtain finer, non-superconducting RE_2BaCuO_5 (RE-211) precipitates [6] in the superconducting phase matrix, either by a chemical route or through irradiation [7, 8]. These techniques generate defects that act as effective flux pinning centres in the bulk superconductor that resist the Lorentz force exerted on flux vortices in the presence of an applied magnetic field, which results directly in increased J_c.

Fabricating bulk, single grains of (RE)BCO is challenging, since the growth of the superconducting RE-123 phase is relatively a complex phenomenon, and depends on several growth parameters. Top seeded melt growth (TSMG) is a well-established processing technique for the fabrication of single grains of (RE)BCO. In general, the starting composition consists of a mixture of RE-123, RE-211 and a small quantity of a grain-refining agent, such as Pt or CeO_2. The RE-123 phase, when heated above its peritectic temperature 'T_p', melts incongruently and decomposes into a solid RE-211 phase and a barium and copper rich liquid phase ($BaCuO_2$ and CuO). These phases recombine with each other on cooling below T_p, to form the superconducting RE-123 phase under appropriate undercooling conditions.

Randomly oriented, multiple grains of RE-123 nucleate and grow in the absence of a seed, which leads to the formation of multi-grain, bulk (RE)BCO. The presence of grain boundaries adversely affects the superconducting properties of the bulk superconductor, and typically reduces J_c by up to 3 orders of magnitude [9]. Hence, it is essential that grain boundaries are eliminated from the bulk (RE)BCO microstructure, which can only be achieved by processing the entire bulk sample in the form of a large, single grain. As a consequence, a combination of novel seeding techniques, with limited undercooling to inhibit homogenous grain nucleation, have been developed and employed over the past 30 years to grow single grains of various (RE)BCO bulk superconductors.

Seed crystals employed in TSMG are required to have the following properties:

(i) A melting temperature significantly above the seeding temperature of the bulk (RE)BCO superconductor to enable the effective nucleation of epitaxial grain growth.
(ii) A similar crystal structure to the superconducting RE-123 phase of the target single grain.
(iii) Phase stability with the Ba–Cu–O melt.

Both Nd-123 ($T_p = 1068$ °C) and Sm-123 ($T_p = 1054$ °C) compounds, which have a T_p higher than Y-123 ($T_p = 1005$ °C), work as effective seed crystals for the TSMG of YBCO. Mg-doped Nd-123 (Mg-NdBCO) generic seed crystals ($T_p = 1083$ °C) grown via a MG technique, on the other hand, have proved to be moderately effective for seeding Nd-123, Gd-123 or Sm-123 [10, 11] and can aid the seeding process of (RE)BCO bulk superconductors, in general [10]. Alternatively, thin films of NdBCO/MgO and NdBCO/YBCO/MgO [12–16] have been used successfully as seed crystals to fabricate large YBCO, SmBCO, NdBCO and GdBCO single grains based on superheating phenomenon. In addition to being expensive, thin film seeds suffer from the serious problem of the diffusion of Mg from the seed crystal substrate into the single grain bulk superconductor [17], which results in possible contamination that reduces J_c locally and hence produces a non-uniform J_c distribution throughout the volume of the sample.

The seeding and single grain growth processes become even more critical when Ag is added to the (RE)BCO precursor composition to improve the mechanical properties of the fully grown bulk superconductor [18]. At the processing temperature, which is typically in excess of 1000 °C, Ag diffuses from the semi-solid melt of the bulk sample into the seed crystal, causing a significant reduction in its T_p, and thereby degrading the properties of the seed. The so-called buffer technique developed recently addresses this problem to some extent [19–22]. These processing challenges are more severe in samples fabricated by infiltration and growth (IG) [23, 24], however, despite significant research effort to date, and single grain growth by IG is not yet completely reliable. A number of bulk (RE)BCO and (RE)BCO–Ag samples that failed to grow in the form of large single grains when fabricated via TSMG and buffer-aided top seeded melt growth (BA-TSMG) processes are shown in figure 1(a).

The infiltration and growth technique has emerged as an alternative method for the fabrication of large, single grain bulk (RE)BCO superconductors [25–32]. The key merits of this technique, compared to conventional TSMG, are that it produces samples of near-net shape geometry that contain significantly reduced porosity in the final large grain microstructure. Unfortunately, successful seeding is more challenging in the IG processing due to the presence of large quantities of the aggressive Ba–Cu–O liquid phase at elevated temperatures. The use of a buffer pellet can overcome seeding problems to certain extent [33, 34], although, due to the fact that the liquid phase is highly reactive, greater attention has to be paid to the composition of the buffer pellet, its geometry (and height in particular) and to the infiltration temperature and time. The bulk samples will fail to grow into single grains if these parameters are not optimised. A selection of (RE)BCO and (RE)BCO–Ag bulk samples fabricated by top seeded infiltration growth (TSIG) that failed to grow in the form of single grains is shown in figure 1(b).

Here we report the reasons for the failure of the various samples to grow in the form of large single grains based on seed crystal considerations and describe procedures by which these failures can be avoided. A robust seeding technique is developed that addresses the long-standing problem of efficient and reliable seeding in (RE)BCO and (RE)BCO–Ag bulk superconductors. The present work extends the applicability of generic seed technology [10, 11, 35] and also addresses the growth of RE–Ba–Cu–O systems that exhibit higher T_p for potential use in a range of high field applications.

2. Experimental

Primary precursor powders of RE-123 and RE-211, each of purity 99.9% and particle size 1–3 μm, were procured from Toshima in order to prepare (RE)BCO and (RE)BCO–Ag bulk superconductors via TSMG, BA-TSMG and BA-TSIG fabrication techniques. Grain-refining agents, such as CeO_2 and Pt were added to the precursor composition, which included Ag_2O for the fabrication of (RE)BCO–Ag.

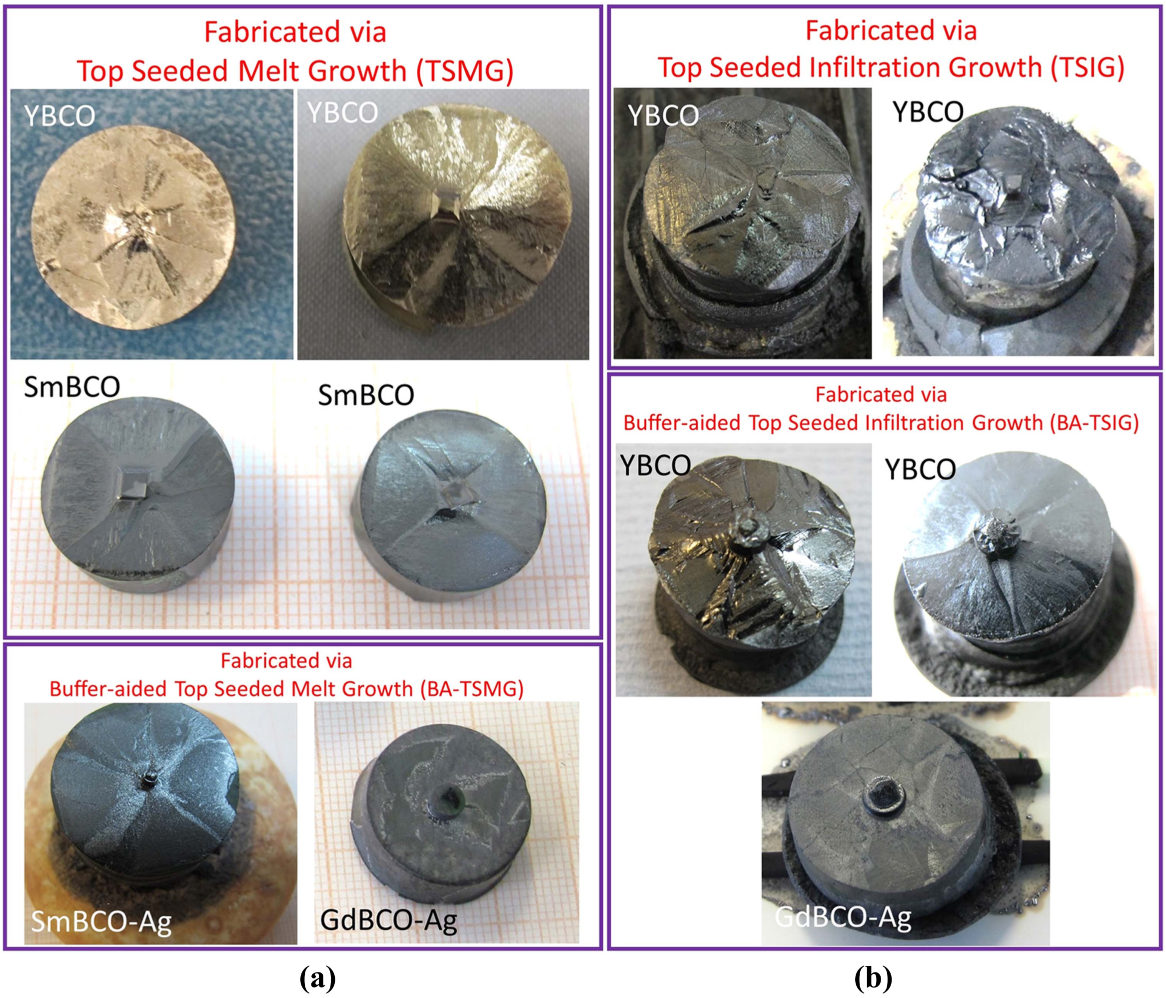

Figure 1. Failed (RE)BCO and (RE)BCO–Ag samples grown by (a) top seeded melt growth (TSMG) and buffer-aided top seeded melt growth (BA-TSMG) and (b) top seeded infiltration growth (TSIG) and buffer-aided top seeded infiltration growth (BA-TSIG) processes.

The TSMG processed (RE)BCO samples were of the composition: 75 wt% RE-123 + 25 wt% RE-211 + 0.5 wt% CeO_2/Pt similar to that discussed in [21]. A Yb-based liquid phase powder was prepared in a molar ratio of Yb_2O_3:CuO:$BaCuO_2 = 1:10:6$ and this was used for preparing the liquid phase reservoir pellets employed to obtaining the samples via TSIG. For sample fabrication via TSIG, the RE-211 preforms enriched with 1 wt% CeO_2 were supported on the Yb-based liquid phase reservoir pellets and subjected to infiltration and subsequent single grain growth as discussed in [34, 36]. Buffer pellets of the composition 75 wt% RE-123 + 25 wt% RE-211 were used to obtain single grains via TSMG and TSIG fabrication routes. The precursor powders were mixed thoroughly using an auto-mixer or Turbula® (Willy A. Bachofen model T2F), and compacted using a uniaxial press.

Several different seed crystals were employed in this study: NdBCO, Mg-doped NdBCO generic seeds fabricated by MG (fabricated following the procedure indicated in [10, 11]), a commercial NdBCO thin film (consisting of a 300 nm thick NdBCO film deposited on 0.5 mm thick MgO substrate, as procured from Ceraco Ceramic Coating GmbH, Germany) and Mg-doped NdBCO generic seeds fabricated by IG (more details of this sample-seed fabrication process can be found in section 3.2 of the present paper). A typical heat treatment process involved in fabricating samples in the present work involved heating the sample assembly to T_{max}, holding the temperature at T_{max} for between 30 min and 1 h to allow the liquid phase to infiltrate into the RE-211 preform, cooling the sample quickly to T_{g1} and then more slowly to T_{g2} (where T_{g1} and T_{g2} represent the growth temperatures) to allow single grain growth to occur. The fully grown samples were oxygenated subsequently in a tube furnace at 460 °C for 200 h under flowing oxygen with a flow rate of 100 ml min^{-1}.

Seed crystals were cleaved from the various melt grown samples, mounted in a cold setting compound and then polished down to 1 μm employing SiC papers and diamond paste for microstructural analysis. The seed microstructures were observed and recorded using a USB microscope (Dinolite) and an optical microscope with an attached polariser (Nikon). TG-DTA measurements were carried out on individual, cleaved seed crystals using a heating and cooling rate of 10 °C min^{-1}.

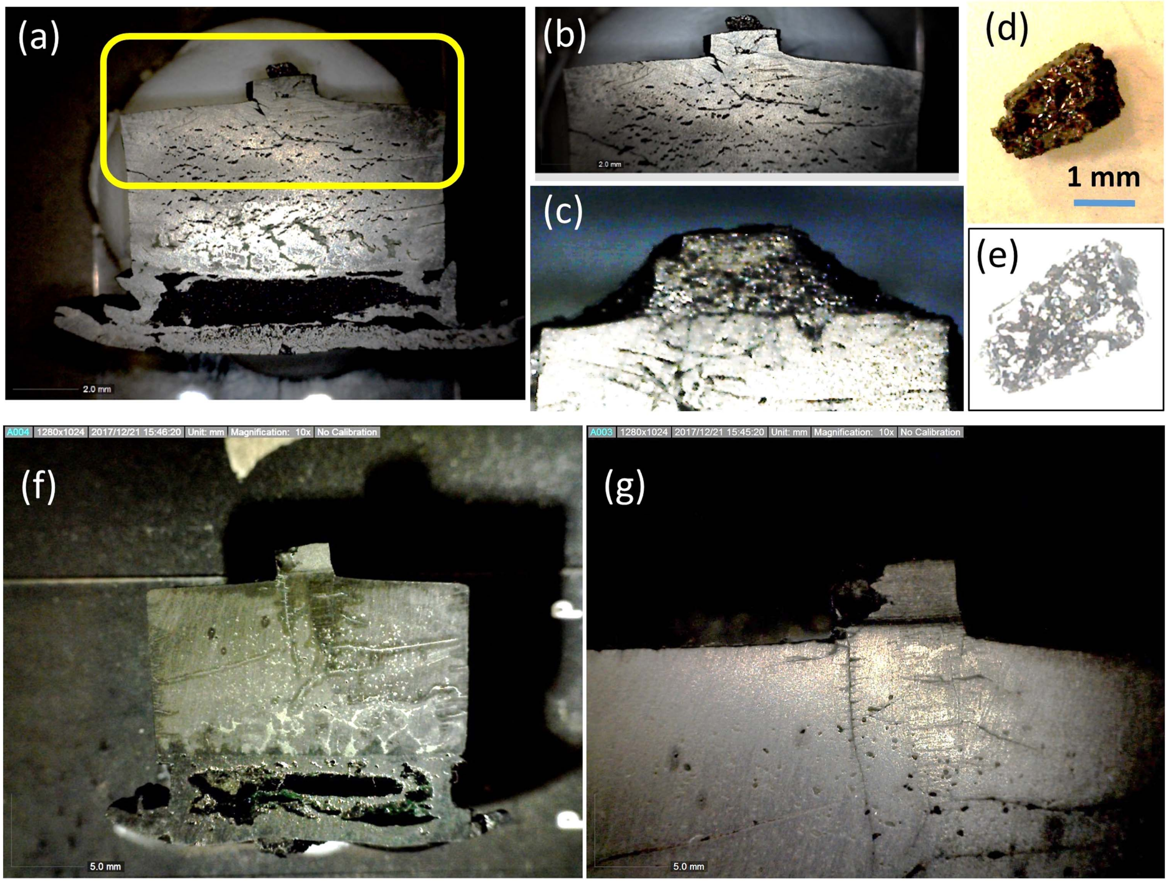

Figure 2. (a) Cross-section of a failed YBCO sample processed via the buffer-aided top seeded infiltration growth technique. (b) and (c) show that the seed crystal failed to seed the buffer pellet, which prevented the sample growing subsequently in the form of a single grain. Failure of the seed due to an accumulation of liquid phase in the pores of the seed crystal is evident in (d) and (e). Another example of failure resulting from the partial dissolution of seed crystal can be seen in (f). A magnified image of the seed/sample interface of the sample in (f) is shown in (g).

3. Results and discussion

3.1. Failure in seeding

Seeding is a critical step for achieving single grain growth of (RE)BCO bulk superconductors. NdBCO or SmBCO cleaved crystals are used typically as seed crystals for seeding the YBCO system. The quality of the seed crystal is critical and, even if the seed is chosen well, the single grain growth process can fail for several reasons. The seeds employed in the TSMG and TSIG processes often dissolve in the RE-211 + liquid mixture formed by the peritectic reaction, even though the processing temperature is well below the melting point, or T_p, of the seed crystal [37]. The partial or complete seed dissolution problem is most severe at the seed/semi-solid melt interface area. The extent of seed dissolution depends on several parameters such as the specific (RE)BCO system being grown, its chemical composition, the maximum processing temperature, the time interval during which the sample assembly is exposed to the maximum processing temperature and the dimensions/thickness of the seed crystal [38–41].

The use of a buffer between the seed and the partial melt can help reduce the failure rate appreciably, although failures still occur under the following conditions:

(a) The maximum processing temperature is close to the seed melting temperature.
(b) The sample assembly is subjected to the maximum processing temperature for a long time.
(c) Excessive amounts of liquid phase come into contact with the seed crystal.

These effects are more critical in the following two cases:

(i) Sample fabrication via TSIG
The highly reactive liquid phase (comprising of $BaCuO_2$ and CuO) that infiltrates from the liquid phase reservoir to the RE-211 preform can easily come into contact and react with the seed crystal, causing partial/complete melting of the seed, and thereby affecting the properties of the seed crystal.
(ii) The fabrication of (RE)BCO–Ag bulk superconductors

Ag from the parent bulk can react easily with the seed crystal during the fabrication of (RE)BCO–Ag bulk superconductors,

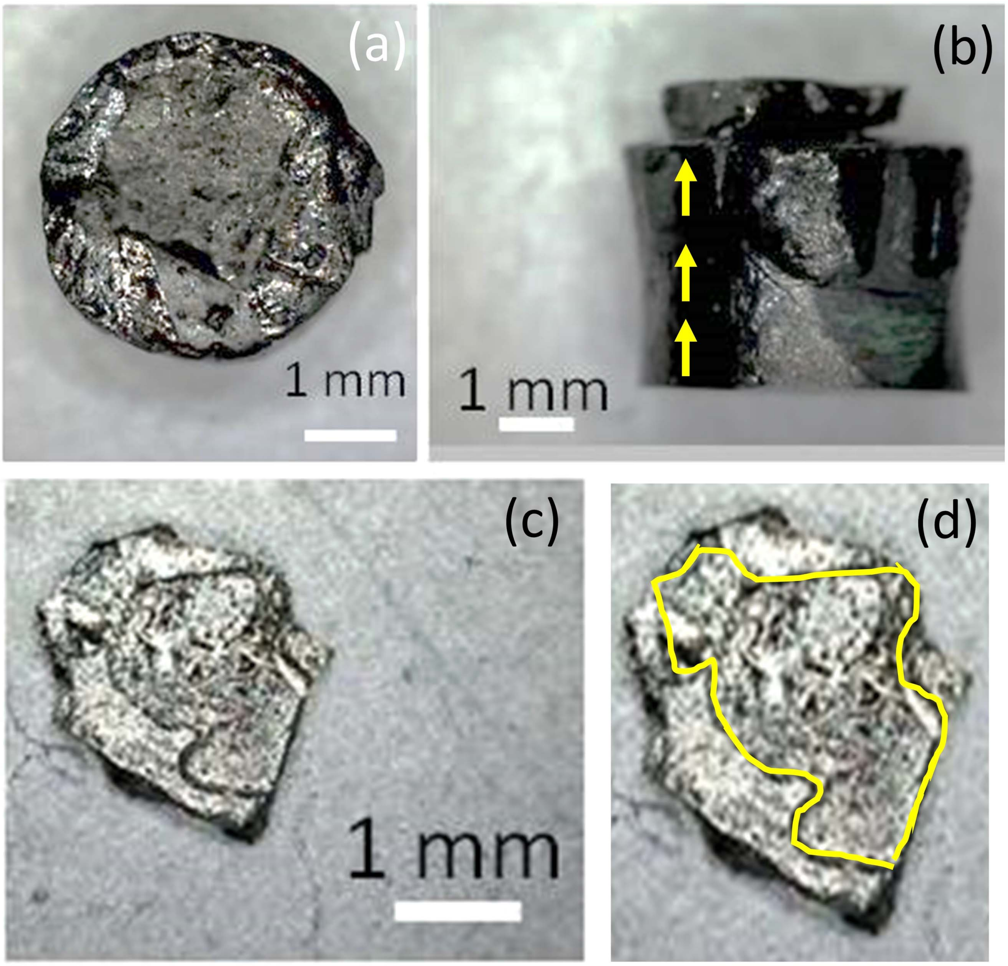

Figure 3. (a) Top and (b) side views of a buffer pellet capped with a NdBCO seed crystal, after removal from a failed sample. Evidence that the seed crystal failed even to seed the small buffer pellet can be seen in (a) and (b) via the creation of sub-grains. The surface of the seed crystal in contact with the buffer pellet was removed from the pellet, and is shown in (c). The liquid phase segregating and contacting the seed crystal can be seen clearly in (c) and is highlighted in (d).

Figure 4. Mg-NdBCO generic seed crystals fabricated via the IG technique.

thereby reducing the melting temperature of the seed crystal significantly, and resulting in the failure of the single grain growth process.

Direct evidence of seed failure observed for TSIG processed YBCO samples are shown in figure 2.

It is evident from figure 2 that the NdBCO seed crystal has been exposed to the liquid phase during processing, causing it to melt partially and prevent the formation of a single grain. In addition, the flat surface of the seed crystal was found to be very irregular after processing. Another example, where partial melting of the seed led to the formation of sub-grains in the buffer pellet itself, can be seen in figure 3. The main sample cannot form a single grain once a sub-grain forms in the buffer pellet. The entry of liquid phase to the seed crystal through the buffer pellet is evident on the contact surface (obtained after removing the seed crystal from the buffer) as shown in figure 3(c).

These observations are in good agreement with the phase diagram for the NdBCO + liquid phase component (comprising of $BaCuO_2$ and CuO) [42, 43]. It is clear from the

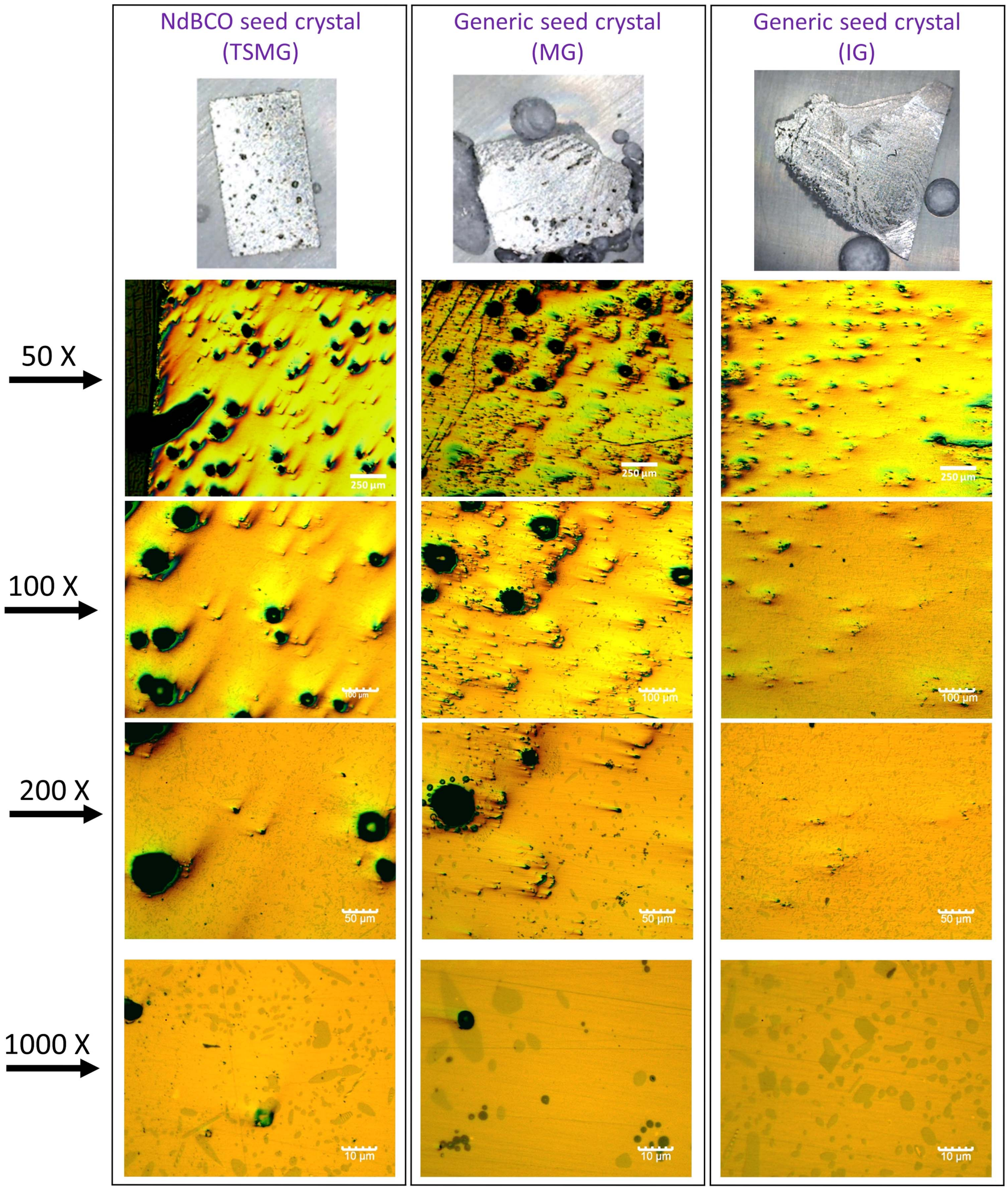

Figure 5. Microstructures of three different seed crystals: (i) NdBCO seed crystal (obtained via TSMG), (ii) Mg-NdBCO generic seed crystal (fabricated via MG) and (iii) Mg-NdBCO generic seed crystal (fabricated via IG). Optical micrographs obtained under different magnifications (50X to 1000X) clearly indicate the extent of porosity in each of these seed crystals.

solubility curves of NdBCO in the BaO/CuO mixture that the NdBCO phase melts at a lower temperature compared to its characteristic T_p [42]. Yao *et al* have reported that NdBCO melts completely in the presence of a large liquid phase component and that no crystallisation is possible when a Nd_2O_3 crucible was used to contain the BaO and CuO liquid phase mixture [42]. This explains clearly the potential of the liquid phase to affect the structural integrity of the NdBCO seed crystal and the associated deterioration of its properties.

3.2. Fabrication of generic seeds via IG

Mg-NdBCO generic seeds were fabricated via the IG technique. For this purpose, Nd-422 powder was compacted into a pellet and supported with Yb-based liquid phase, with a MgO used to seed the bulk samples. The sample assembly was heated to 1025 °C and held at this temperature of 1 h to enable infiltration of liquid phase into the Nd-422 preform. The arrangement was heated subsequently to 1125 °C and held at this temperature for 30 min followed by slow cooling to

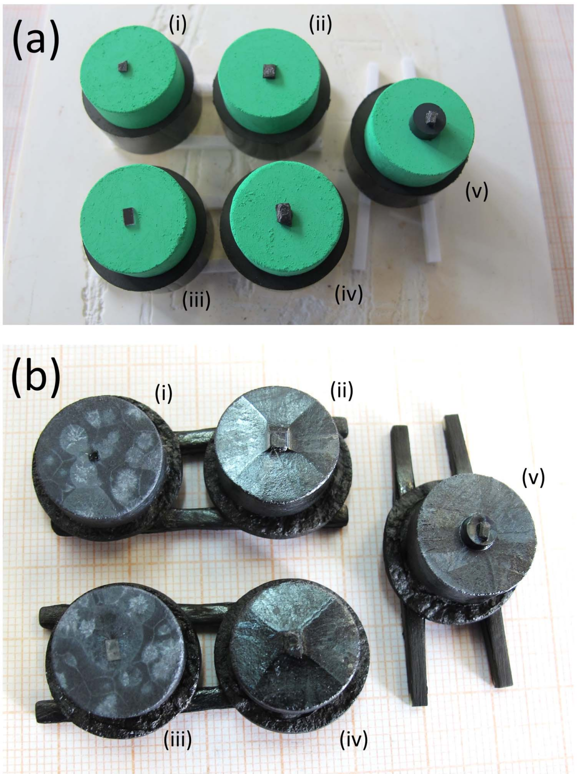

Figure 6. YBCO samples fabricated via top seeded infiltration growth (TSIG) and buffer-aided TSIG for different choices of seed crystal. The sample arrangements for TSIG and BA-TSIG are shown in (a). The fully processed samples are shown in (b). The stability of (i) the NdBCO seed, (ii) the generic seed fabricated by MG, (iii) the NdBCO thin film seed (iv) the generic seed fabricated by IG and (v) the NdBCO seed with buffer pellet, were investigated as part of this study.

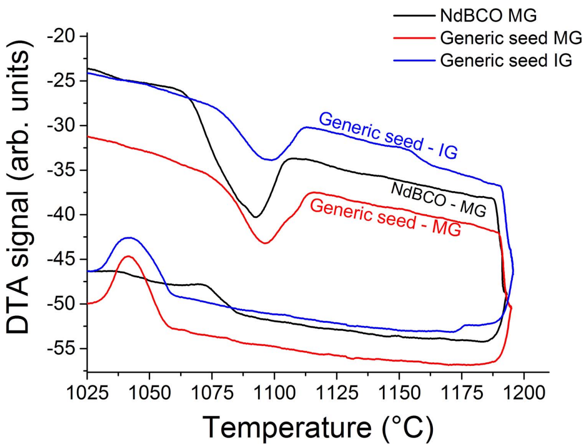

Figure 7. DTA results obtained for the three seed crystals investigated: (i) the NdBCO seed, (ii) the generic seed fabricated by MG and (iii) the generic seed fabricated by IG. The heating and cooling rates were 10 °C min^{-1} in each of these measurements.

1080 °C, before furnace cooling to room temperature. The as-grown IG processed generic seeds are shown in figure 4. The grains with shiny surfaces apparent in the figure are the generic seed crystals, which were subsequently cleaved from these bulk samples.

3.3. A comparison of different potential seeds

The properties of three different seed crystals: (i) NdBCO, (ii) Mg-NdBCO (the generic seed) fabricated by MG and (iii) Mg-NdBCO fabricated by IG were compared by investigating in detail their microstructural and thermal properties. Microstructural comparison of the three seed crystals is shown in figure 5. Optical micrographs obtained under different magnifications (between 50X and 1000X) for each of the three seed crystals are presented in the figure.

It can be seen from figure 5 that the porosity in each of the three seed crystals is very different. NdBCO (fabricated via TSMG) has the highest porosity whereas the generic seed (fabricated via IG) has the least. It can be seen that the liquid phase originating from either the liquid phase reservoir or the RE-123 phase actually accumulates within the pores of the seed crystals. This reacts subsequently with localised regions of the seed, which results in partial melting of the seed crystal. This can subsequently cause sub-grains to form in the main sample due to the partial/complete melting of the seed, resulting in a seed crystal that is not aligned uniformly with the local crystallographic orientation of the RE-123 phase in the body of the bulk superconductor. The fact that the generic seed fabricated by IG contains fewer and smaller sized pores reduces this problem dramatically, which explains why the success rate of the growth of single grains from IG processed generic seeds is improved appreciably.

In order to further examine this aspect of the melt process, a set of samples were chosen for fabrication via TSIG with different seeds in the configurations and conditions where seed melting would normally be expected to occur. The sample assemblies were prepared and capped with different potential seed crystals: NdBCO by TSMG, generic seed fabricated by MG, a commercially procured NdBCO thin film seed, a generic seed fabricated by IG and a NdBCO seed with a buffer. The sample arrangements before and after melt processing are shown in figures 6(a) and (b), respectively.

It can be seen from the images in figure 6 that the NdBCO seed and the NdBCO thin film seed have melted completely during the high temperature processing stage. This is due to the fact that the liquid phase can easily come into contact with the seed crystal during TSIG. On the other hand, both the generic seeds (fabricated via MG and IG) successfully resisted reaction with the liquid phase component, which enabled heterogeneous nucleation and single grain growth for YBCO. It can be seen that contact of the liquid phase with the buffer pellet is minimised to some extent for the NdBCO seed crystal, although a small amount of liquid phase still reaches the NdBCO seed and reacts with it causing a partial melting, leading to multiple grain formation, as can be seen in figure 6(b)-(v).

The melting temperature of each of the seed crystals investigated was measured using TG-DTA and the results obtained are shown in figure 7. It can be seen that both generic seeds have a higher melting temperature, which is consistent with earlier reports of the properties of generic seeds [10, 11]. Furthermore, the generic seed processed by IG

Figure 8. (RE)BCO and (RE)BCO–Ag (where RE = Y, Gd and Sm) samples fabricated successfully via a buffer-aided top seeded infiltration growth (BA-TSIG) process employing a Mg-NdBCO generic seed crystal fabricated by IG. A GdBCO-Ag sample fabricated via BA-TSMG employing the generic seed crystal fabricated by IG is also shown.

exhibits even better stability due to its relatively dense microstructure.

3.4. Robust seeding methodology

The use of a buffer between the seed and the grain has been shown to protect the seed crystal by minimising infiltration of the liquid phase and, therefore, avoiding melting of the seed crystal. In addition, the buffer pellet prevents the propagation of any defects that may be generated at the seed/sample interface due to lattice mismatch effects [21]. In the present study, we observe that Mg-NdBCO generic seeds fabricated by IG are the most effective. It was decided, therefore, to investigate the relative benefits of a combination of the use of a Mg-NdBCO generic seed fabricated by IG and the use of a buffer for preventing seeding failure. The approach of seed buffering has been employed and tested on more complex systems, such as GdBCO, GdBCO-Ag and SmBCO fabricated by TSIG and top seeded melt growth techniques, as shown in figure 8. Every attempt to fabricate samples by this combined technique to date have been successful.

This investigation has demonstrated clearly that the robust seeding technique can make the seeding process much more reliable, thereby enabling easier and successful fabrication of large grain (RE)BCO and (RE)BCO–Ag bulk superconductors for practical applications.

4. Conclusions

The seeding of large grain, bulk (RE)BCO superconductors is a key step in their reliable fabrication. The majority of the failures in the fabrication of these materials can be attributed to seed melting due to the liquid phase, either initiated in the sample as a result of incongruent melting of the RE-123 phase or supplied from a liquid phase reservoir, coming into direct contact with the seed crystal. Seed melting results invariably in the formation of multiple grains within the bulk superconductor. We have shown that the presence of large pores in the seed crystal is one parameter that causes seed failure, since they form regions where the liquid phase segregates and may come into contact with the seed crystal for an extended period during the growth process. Significantly, this is the first time the porosity of the seed crystal has been studied as part of the melt process. To overcome this problem, Mg-doped NdBCO generic seeds were fabricated by the infiltration growth technique. These seeds exhibit a relatively dense microstructure with greatly reduced porosity compared to conventional seed crystals fabricated by MG. This is attractive from a processing point of view, since the liquid phase cannot accumulate in the pores of the seed crystal. Furthermore, the combination of a low porosity generic seed crystal fabricated by IG and the buffer-aided technique has led directly to the development of a robust seeding technique for the fabrication of (RE)BCO and (RE)BCO–Ag bulk superconductors. This, in turn, has led to the successful fabrication of YBCO single grains and more challenging and complicated (RE)BCO systems such as SmBCO, YBCO-Ag, GdBCO and GdBCO-Ag by the top seeded infiltration and growth technique, where the presence of an aggressive liquid phase is known to be extremely problematic for effective seeding. The combination of the use of seeds grown by IG and the use of buffer pellets together expands greatly the range of conditions under which successful seeding of large, single grains can take place. We expect this to be of significant benefit to both the laboratory-based and commercial production of large, single grain bulk superconductors for high field applications.

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