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# Control of Y-211 content in bulk YBCO superconductors fabricated by a buffer-aided, top seeded infiltration and growth melt process

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## Abstract

Bulk (RE)–Ba–Cu–O ((RE)BCO, where RE stands for rare-earth), single grain superconductors can trap magnetic fields of several tesla at low temperatures and therefore can function potentially as high field magnets. Although top seeded melt growth (TSMG) is an established process for fabricating relatively high quality single grains of (RE)BCO for high field applications, this technique suffers from inherent problems such as sample shrinkage, a large intrinsic porosity and the presence of (RE)<sub>2</sub>BaCuO<sub>5</sub> (RE-211)-free regions in the single grain microstructure. Seeded infiltration and growth (SIG), therefore, has emerged as a practical alternative to TSMG that overcomes many of these problems. Until now, however, the superconducting properties of bulk materials processed by SIG have been inferior to those fabricated using the TSMG technique. In this study, we identify that the inferior properties of SIG processed bulk superconductors are related to the presence of a relatively large Y-211 content (~41.8%) in the single grain microstructure. Controlling the RE-211 content in SIG bulk samples is particularly challenging because it is difficult to regulate the entry of the liquid phase into the solid RE-211 preform during the infiltration process. In an attempt to solve this issue, we have investigated the effect of careful control of both the infiltration temperature and the quantity of liquid phase powder present in the sample preforms prior to processing. We conclude that careful control of the infiltration temperature is the most promising of these two process variables. Using this knowledge, we have fabricated successfully a YBCO bulk single grain using the SIG process of diameter 25 mm that exhibits a trapped field of 0.69 T at 77 K, which is the largest value reported to date for a sample fabricated by the SIG technique.

**Keywords:** bulk superconductor, (RE)BCO, infiltration and growth, control of RE-211, flux pinning, trapped field, YBCO

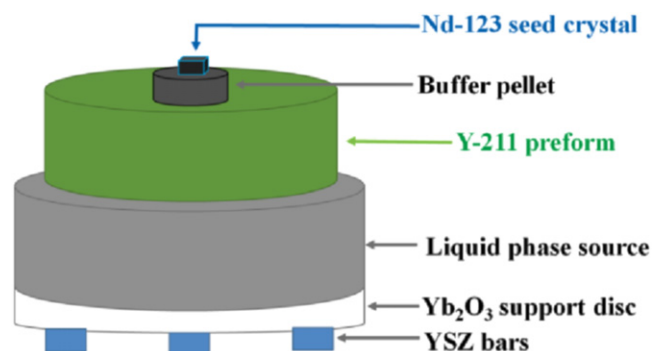
(Some figures may appear in colour only in the online journal)

## 1. Introduction

Bulk high temperature superconductors fabricated in the form of large, single grains can potentially be used for a variety of applications ranging from trapped field magnets, with performance far in excess of normal material, to compact electric motors and friction free self-stabilising bearings for energy



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**Figure 1.** Schematic illustration of the sample arrangement, comprising a Y-211 preform capped with a buffer pellet and a NdBCO seed crystal, a liquid phase reservoir and a  $\text{Yb}_2\text{O}_3$  support disc.

storage flywheels [1, 2]. The ultimate performance of these materials is defined typically by their superconducting properties, including maximum trapped field and critical current density ( $J_c$ ). A large  $J_c$  may be achieved by the enhancement of flux pinning by ensuring the presence of an optimum amount of unreacted  $(\text{RE})_2\text{BaCuO}_5$  (RE-211, where RE stands for rare-earth) second phase material in the superconducting  $(\text{RE})\text{Ba}_2\text{Cu}_3\text{O}_{7-x}$  (RE-123) phase matrix. Significantly, a fine particle size of any unreacted RE-211 phase correlates directly to increased pinning with increasing defect density and as the defect size becomes a better match to the flux vortex core size, which is responsible directly for an increase in  $J_c$  [3].

The top seeded melt growth process (TSMG) is a well-established technique for the growth of high performance, single-grain (RE)BCO bulk superconductors [4–7]. A practical example of the remarkable properties of single grain superconductors fabricated by this technique is the generation of a trapped magnetic field of 17.6 T by a bulk superconductor at 26 K [8]. In order to achieve very large trapped fields, therefore, it is necessary to fabricate large, single-grain (RE)BCO bulk superconductors with microstructures that can support large critical currents.

Although the TSMG approach is well established, it exhibits a number of intrinsic problems that, ultimately, limit the field generating potential of bulk superconductors. These include the considerable shrinkage of the final bulk sample that occurs during TSMG processing, the presence of a large amount of porosity and regions that are free of the RE-211 phase in the fully processed sample microstructure [9, 10]. These defects are due typically to the outflow of liquid phase during heat treatment and to the generation of oxygen gas and the conditions under which the green pellets are commonly prepared (open air pressing). Recently, a number of researchers have explored the seeded infiltration and growth (SIG) process as a practical alternative that does not exhibit some of the less desirable features of TSMG [9–16].

In the conventional SIG process, a RE-211 preform capped with a seed crystal is placed in contact with a liquid phase reservoir and the whole arrangement is subjected to an appropriate thermal process. The Cu-rich liquid phase

(comprising of  $\text{BaCuO}_2$  and  $\text{CuO}$ ) originates from the liquid phase reservoir during the SIG process, which then infiltrates into a RE-211 preform, with which it reacts subsequently to form the RE-123 phase. At this stage, heterogeneous nucleation of the RE-123 phase is initiated by the seed crystal, prior to its growth into a large, single grain. To date, the superconducting properties exhibited by infiltration grown samples have generally been inferior to samples fabricated by the TSMG process [17, 18].

A possible reason for the observed inferior performance of samples processed by SIG has been investigated and discussed. Two approaches to overcome these problems have been investigated and compared, and the most promising is established from microstructural considerations. A single grain YBCO sample of diameter 25 mm diameter fabricated using this approach exhibits improved superconducting properties, including the highest value of trapped field at 77 K in a SIG sample reported to date.

## 2. Experimental details

Commercial Y-123 (3N, Toshiba), Y-211 (3N, Toshiba),  $\text{CeO}_2$  (3N, Alfa Aesar),  $\text{Yb}_2\text{O}_3$  (3N, Alfa Aesar),  $\text{Ba}_3\text{Cu}_5\text{O}_8$  (3N, Toshiba) and  $\text{BaO}_2$  (95% Sigma Aldrich) powders were used to develop the SIG process in the present study. Recent work [18] has found that a Yb-based liquid phase leads to better and more reliable grain growth and, consequently, this approach was adopted in this investigation.

The Yb-based liquid phase was prepared by mixing together powders of  $\text{Yb}_2\text{O}_3$ ,  $\text{CuO}$  and  $\text{BaCuO}_2$  in a molar ratio of 1:6:10 [19] for 2 h using a Turbula mixer (Willy A. Bachofen model T2F) and agate balls as the milling medium. The liquid phase and Y-211 preform pellets were prepared under an applied uniaxial pressure of 80 MPa using a steel die. This liquid phase reservoir was supported by a  $\text{Yb}_2\text{O}_3$  substrate to prevent loss of liquid phase during processing. The so-called seed-buffering technique [7, 20], was also used in the present study. This involved placing a buffer pellet of composition 75 wt% Y-123 + 25 wt% Y-211 between the seed crystal (cleaved NdBCO) and the Y-211 preform. A schematic of the entire sample arrangement is shown in figure 1.

Each sample assembly (figure 1) was processed using a conventional box furnace. The thermal profile consisted of first heating the sample to 1054 °C and holding for 1 h to enable the liquid phase to form in the reservoir and then to infiltrate into the porous Y-211 preform. The sample was then cooled rapidly at 50 °C h<sup>−1</sup> to 1010 °C after the infiltration step, and then slow cooled at 0.8–0.5 °C h<sup>−1</sup> to 980 °C to ensure heterogeneous nucleation and growth of the Y-123 phase. Finally, each sample was furnace cooled to room temperature. The fully-grown single grain sample was oxygenated for 150–200 h in flowing oxygen with a flow rate of 100 ml min<sup>−1</sup>. The TSIG processed samples required a comparatively longer time for oxygenation compared with samples processed by TSMG due to their relatively dense microstructures [21]. The present sets of samples, therefore,

were oxygenated for optimum, but different, amounts of time under flowing oxygen.

The top surfaces of the oxygenated samples were polished using SiC paper (800 grit) in order to obtain flat surface required for trapped field measurements. Each sample was field cooled to 77 K in an electromagnet at an applied magnetic field of 1.2 T, with  $B$  applied parallel to its  $c$ -axis. The applied magnetic field was then removed and the trapped magnetic flux density profile on the top surface of the sample measured using both hand-held and scanning Hall systems comprising of an array of 19 Hall probes spaced uniformly over the active area of the probe. The air gap between the sample surface and the Hall probe array was maintained constant at approximately 0.8 mm for each separate measurement.

Sections of the fully processed YBCO single grains were prepared using a diamond saw and a mechanical polisher in order to perform microstructural studies and to measure magnetisation hysteresis ( $M$ – $H$ ) loops of the samples. The microstructures were examined using an optical microscope equipped with a polariser. A SQUID magnetometer was employed to record  $M$ – $H$  loops at 77 K in a field of up to 6 T applied parallel to  $c$ -axis for specimens extracted from different locations within each parent YBCO single grain. Current densities were estimated from the width of the  $M$ – $H$  loops using extended Bean's critical state model [22].

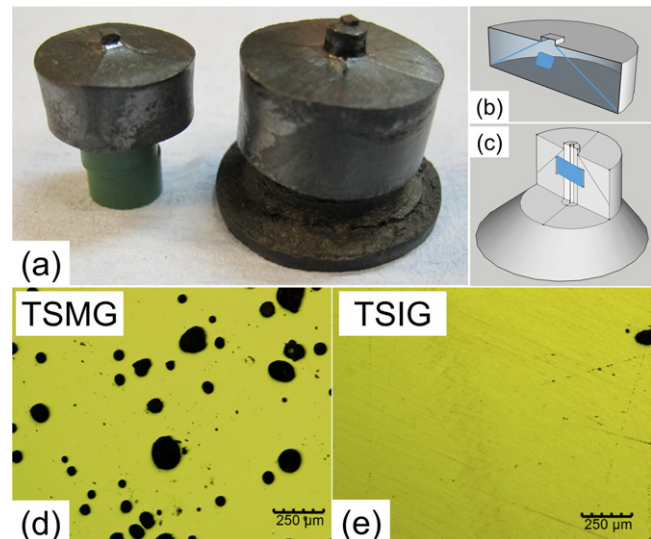
### 3. Results and discussion

#### 3.1. YBCO reference sample fabricated by conventional SIG

A single grain, bulk YBCO sample of diameter 25 mm was fabricated as a reference sample by employing the BA-SIG process. For comparison, a second bulk YBCO single grain was fabricated using a green pellet of similar size by a conventional TSMG technique from a precursor pellet of composition Y-123 + 25 wt% Y-211 + 0.5 wt% CeO<sub>2</sub> pressed uniaxially in a steel die under a pressure of 80 MPa. Further details of the TSMG sample preparation can be found elsewhere [7]. Photographs of these two, single grain samples and their corresponding microstructures (i.e. from the central region of the single grains) are shown in figure 2.

It can be seen from figure 2(a) that SIG processing overcomes the problem of shrinkage, which is unavoidable with the TSMG technique. Furthermore, the microstructure of the SIG processed sample is significantly denser, and without the presence of large pores that are evident in the sample processed by TSMG (see figures 2(d) and (e)). The superconducting properties, including trapped field and field-dependent  $J_c$ , were measured for each single grain sample at 77 K, with the results shown in figure 3.

It can be seen from figures 3(a) and (b) that both the YBCO samples (one fabricated using TSMG and the other by BA-SIG) were fabricated in the form of a single grain. However, the magnitude of the trapped field measured in the YBCO sample processed by TSMG is far superior compared to that fabricated by BA-SIG. The field dependence of  $J_c$  was



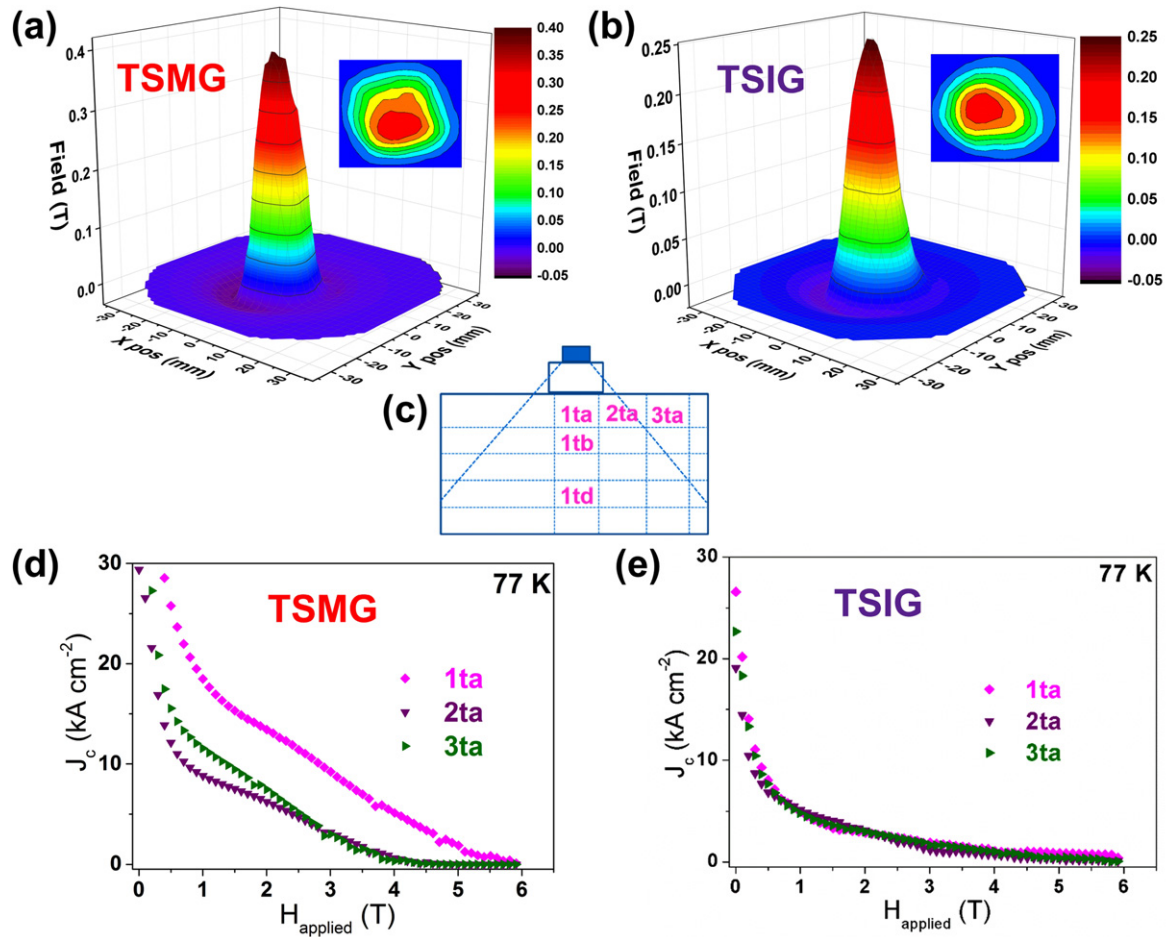
**Figure 2.** (a) Photographs of two YBCO samples: one fabricated employing the TSMG technique and the other employing the BA-SIG technique. The size of the preform pellets was the same in both cases (25 mm in diameter). The positions from where the micrographs are obtained are shown schematically in (b) and (c). Optical micrographs obtained under low magnification (50 $\times$ ) indicating the amount of porosity in each of these samples (processed by TSMG and SIG) can be seen in (d) and (e), respectively.

also measured in both these samples at locations within the parent single grain indicated schematically in figure 3(c).  $J_c(H)$  obtained at 77 K for specimens extracted from the TSMG and BA-SIG processed YBCO single grains are shown in figures 3(d) and (e), respectively. It can be seen that  $J_c(H)$  obtained at different locations of the SIG processed YBCO sample is more uniform compared to that fabricated by TSMG. However, the magnitude of  $J_c$  of the SIG sample is clearly inferior to that observed for the sample fabricated by TSMG.

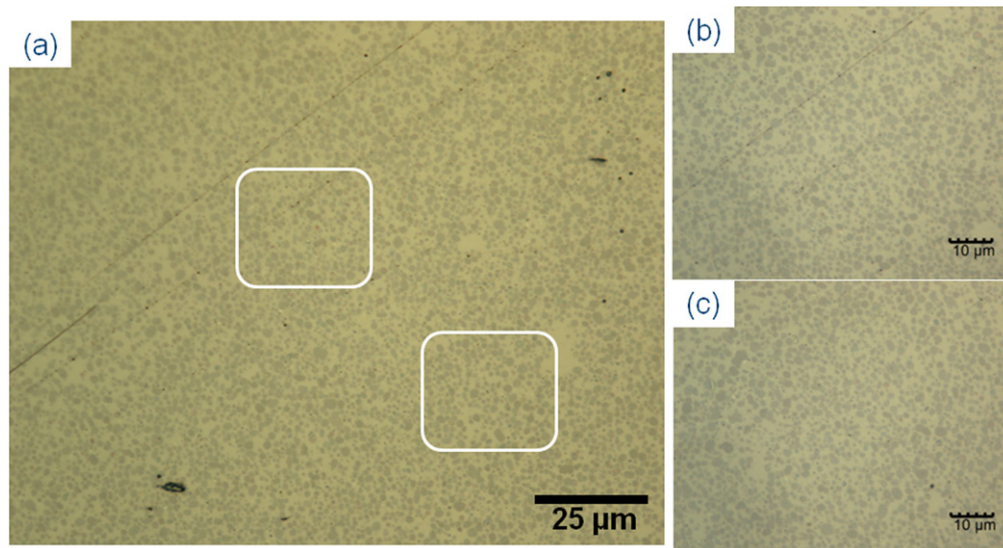
The inferior superconducting properties observed in the SIG processed sample are the motivation for the present study. The apparently superior microstructures achieved in the single grain obtained in the BA-SIG process should reasonably be expected to yield superior superconducting properties, although this is clearly not the case. As a result, a detailed microstructural analysis was performed on these samples to investigate the underlying reasons. Optical micrographs recorded under higher magnifications for the BA-SIG processed YBCO sample (from the central region of the single grain, as shown schematically in figure 2(c)) are shown in figure 4.

It can be seen from figure 4 that refined, Y-211 inclusions are distributed homogeneously throughout the Y-123 matrix, which is encouraging from a flux pinning point of view. Image-J analysis software was used to analyse the amount of Y-211 at several locations of the YBCO sample, from which it was observed that the average Y-211 content in the sample was ~41.8%, which is perhaps too large for optimally enhanced flux pinning. In the literature, it is reported generally that ~25–28 wt% of Y-211 content is optimum for

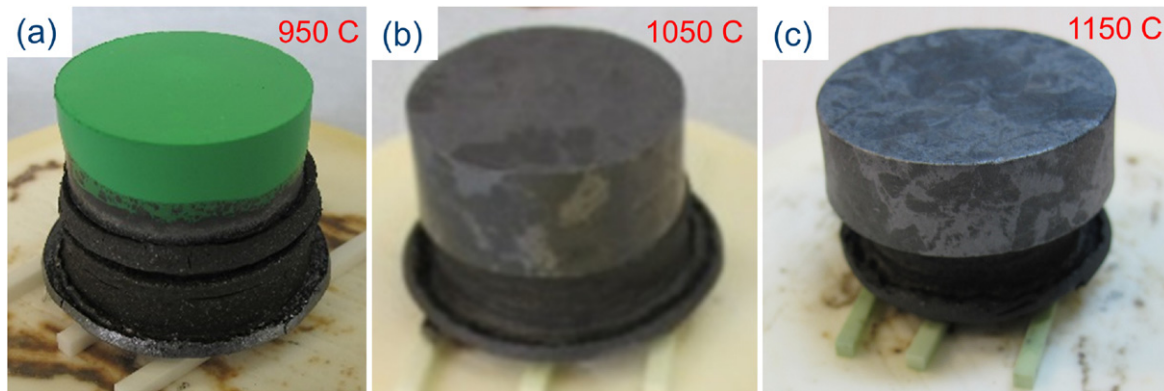




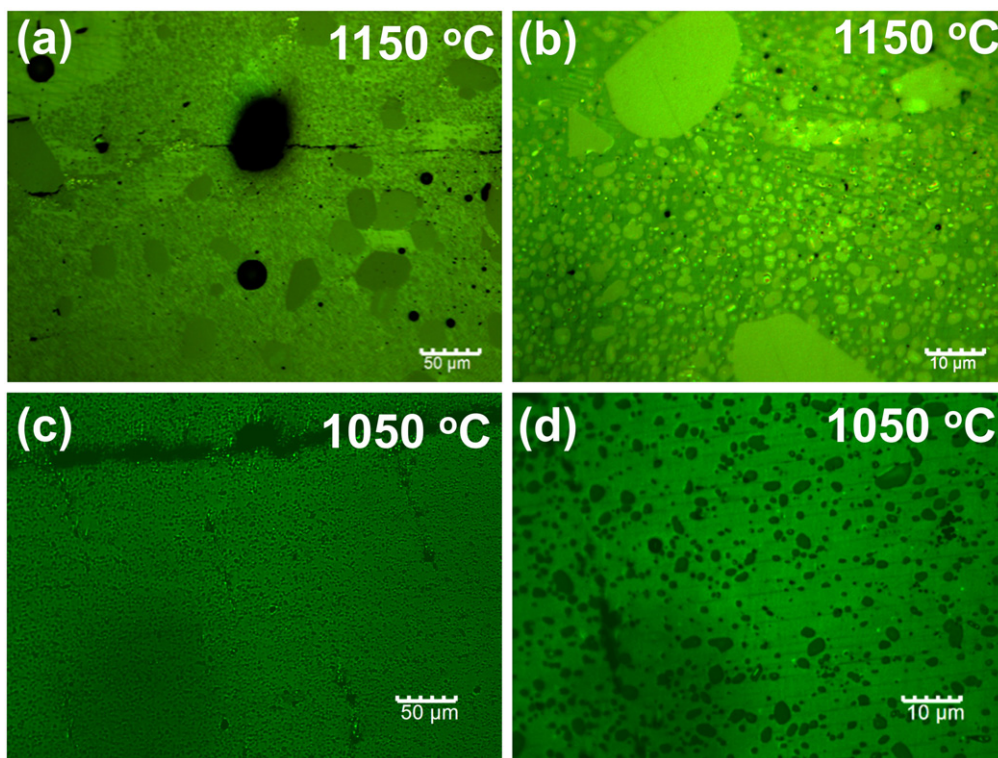
**Figure 3.** A comparison of the superconducting properties of single grain YBCO reference samples at 77 K fabricated by TSMG and TSIG. (a) and (b) show the trapped field and (d) and (e) show the field-dependent critical current densities. A schematic diagram indicating the positions of the specimens within the parent single grain is shown in (c).



**Figure 4.** Optical micrographs recorded for the BA-SIG processed, single grain YBCO sample. The micrograph in (a) was recorded under low magnification (500 $\times$ ). Images obtained under higher magnification (1000 $\times$ ) at the two different locations indicated in (a) are shown in (b) and (c).



**Figure 5.** Samples obtained after the infiltration of liquid phase at (a) 950 °C (b) 1050 °C and (c) 1150 °C. The infiltration time was kept constant at 1 h for each process.



**Figure 6.** Optical micrographs obtained under magnifications of 500 $\times$  and 1000 $\times$ , for the samples processed using an infiltration temperature of 1150 °C (a) and (b) and 1050 °C (c) and (d).

achieving efficient flux pinning [23–25]. Hence, it became evident from this observation that it is necessary to control the amount of Y-211 in the fully processed sample. Two different approaches were investigated in an attempt to achieve this aim:

1. Control of the amount of liquid phase that can enter the Y-211 preform during the infiltration process and;
2. Addition of extra liquid phase in the Y-211 preform prior to SIG processing.

The results obtained from these approaches are described in sections 3.2 and 3.3, respectively.

### 3.2. Effect of variation of infiltration temperature

It is desirable to introduce more liquid phase into the Y-211 preform during heat treatment since the Y-211 content in the final microstructure of a YBCO sample fabricated by BA-SIG process is large (~41.8%). Values of infiltration temperature  $T_i$  of 950 °C, 1050 °C and 1150 °C were used in order to investigate whether the amount of liquid phase entering the Y-211 preform could be controlled by SIG processing. The infiltration time was kept constant at 1 h in each experiment to ensure an objective comparison. The three sample assemblies obtained after the infiltration step at these temperatures are shown in figure 5.



**Table 1.** Amounts of different liquid phases added to the Y-211 preform prior to SIG processing.

Amount of liquid phase ( $x$ in wt%) added to Y-211 preform prior to SIG processing				
Liquid phase	Y-123	Ba <sub>3</sub> Cu <sub>5</sub> O <sub>8</sub>	Yb-based LP	Y-based LP
$x$	10	10	10	10
$x$	20	20	20	20

It can be seen from figure 5 that the infiltration process was not complete for a  $T_i$  of 950 °C. In this case, the liquid phase generated in the liquid phase reservoir could not infiltrate completely into the Y-211 preform, as can be seen clearly in figure 5(a). On the other hand, a  $T_i$  of 1150 °C is probably too high given that the as-processed microstructure for this sample contained coarse Y-211 particles, as can be seen in figure 6(a) and (b).

A  $T_i$  of 1050 °C appears to be optimum from these results, leading to sufficient liquid phase infiltration and resulting in a homogeneous distribution of well-refined Y-211 particles with uniform size in the Y-123 phase matrix. The resulting Y-211 content of ~31% in this sample is very close to the optimum reported for samples fabricated by TSMG. Therefore, by adjusting the infiltration temperature, it is possible to control the amount of liquid phase entering into the RE-211 preform, which, in turn, can be used to tune and adjust the Y-211 content in the fully processed single grain.

**Table 2.** Amount of Y-211 remaining after IG processing.

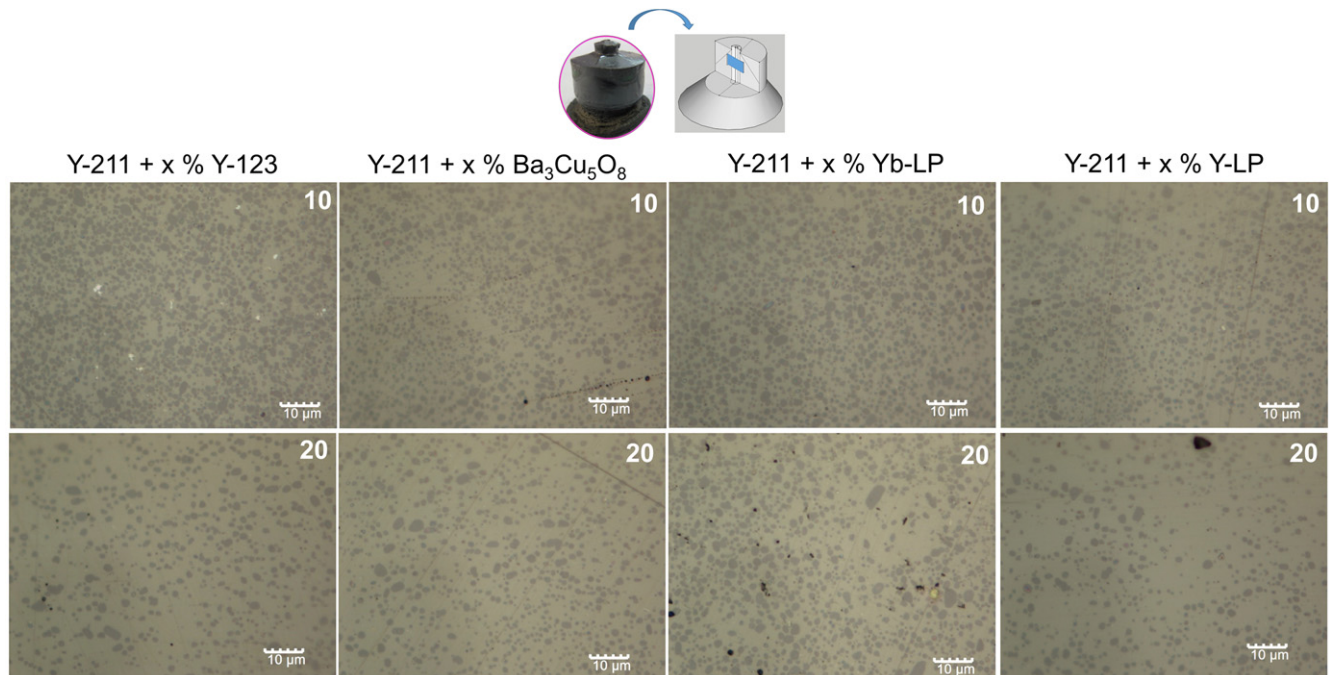
Liquid phase	Y-123	Ba <sub>3</sub> Cu <sub>5</sub> O <sub>8</sub>	Yb-based LP	Y-based LP
$x$ :10	38.87	30.29	31.82	28.17
$x$ :20	16.97	18.73	22.98	16.28

### 3.3. Provision of extra liquid phase prior to SIG

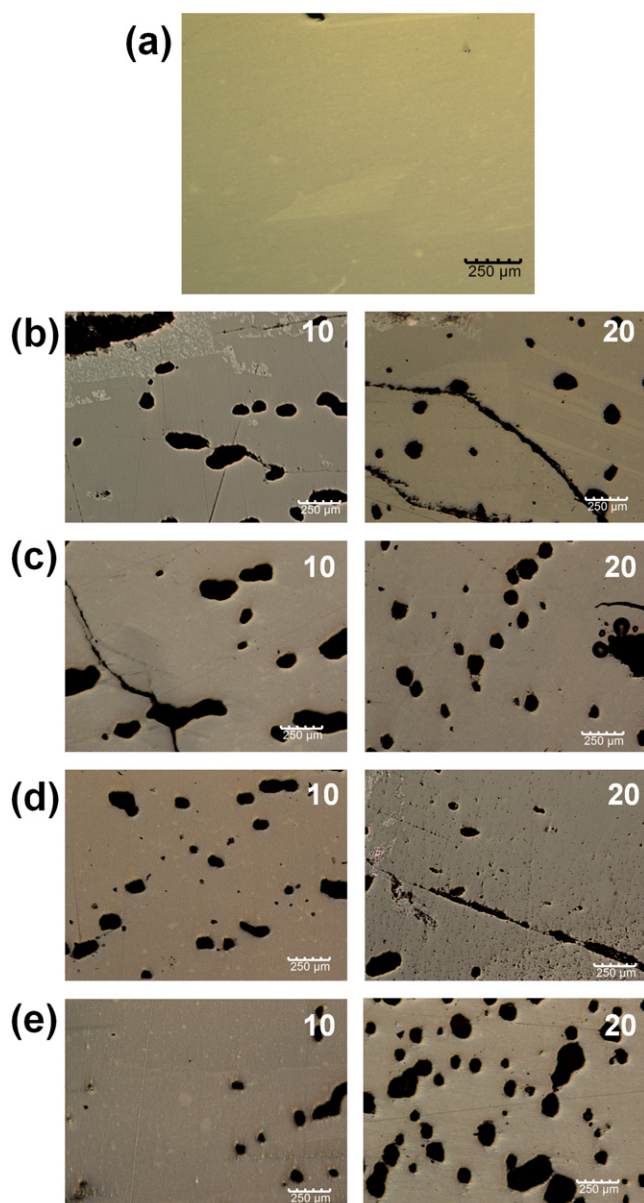
An alternative approach was investigated in order to reduce the Y-211 content in the single grain sample based on the introduction of small amounts of liquid phase powder to the Y-211 preform prior to SIG processing. For this purpose, 10 and 20 wt% (with respect to the Y-211 preform) of different liquid phase (Y-123, Ba<sub>3</sub>Cu<sub>5</sub>O<sub>8</sub>, Yb-based liquid phase, Y-based liquid phase) powders were added to the Y-211 preform prior to SIG processing, as summarised in table 1.

YBCO samples with the composition indicated in table 1 were fabricated by the BA-SIG process using the heat treatment profile described in the experimental section. Each fully processed sample was sectioned through its thickness and polished using SiC paper and then 1  $\mu$ m diamond paste. Optical micrographs under a magnification of 1000 $\times$  were recorded at the centre of each sample, as shown in figure 7.

It can be seen that there is a systematic reduction of Y-211 content with the amount of liquid phase powders added prior to SIG processing. Image-J software was used to analyse the Y-211 content in each of the micrographs, with the amount of Y-211 recorded in each case given in table 2.



**Figure 7.** Schematic illustration of the location within the parent single grain where the optical micrographs were recorded. Microstructures were recorded for YBCO samples in which the starting composition of the preform was Y-211 +  $x$ % liquid phase (Y-123/Ba<sub>3</sub>Cu<sub>5</sub>O<sub>8</sub>/Yb-based LP/Y-based LP) with  $x = 10, 20$ .



**Figure 8.** Low magnification ( $50\times$ ) images obtained in BA-SIG processed YBCO samples (a) for an optimised infiltration temperature of  $1050\text{ }^{\circ}\text{C}$ . (b)–(e) Micrographs corresponding to different amounts (10 and 20 wt%) of liquid phase powders (Y-123,  $\text{Ba}_3\text{Cu}_5\text{O}_8$ , Yb-based liquid phase, Y-based liquid phase) added to the Y-211 preform prior to SIG processing.

### 3.4. Comparison of results

Both attempts to improve the properties of the samples fabricated by SIG reported in the present work enabled the Y-211 content in the fully YBCO BA-SIG processed single grain to be controlled. However, the microstructural study carried out on the samples obtained by both approaches indicated that control of the infiltration temperature is more effective than the addition of liquid phase powders to Y-211 preforms prior to SIG processing. Optical micrographs obtained under lower magnification ( $50\times$ ) for all these YBCO samples are shown in figure 8.

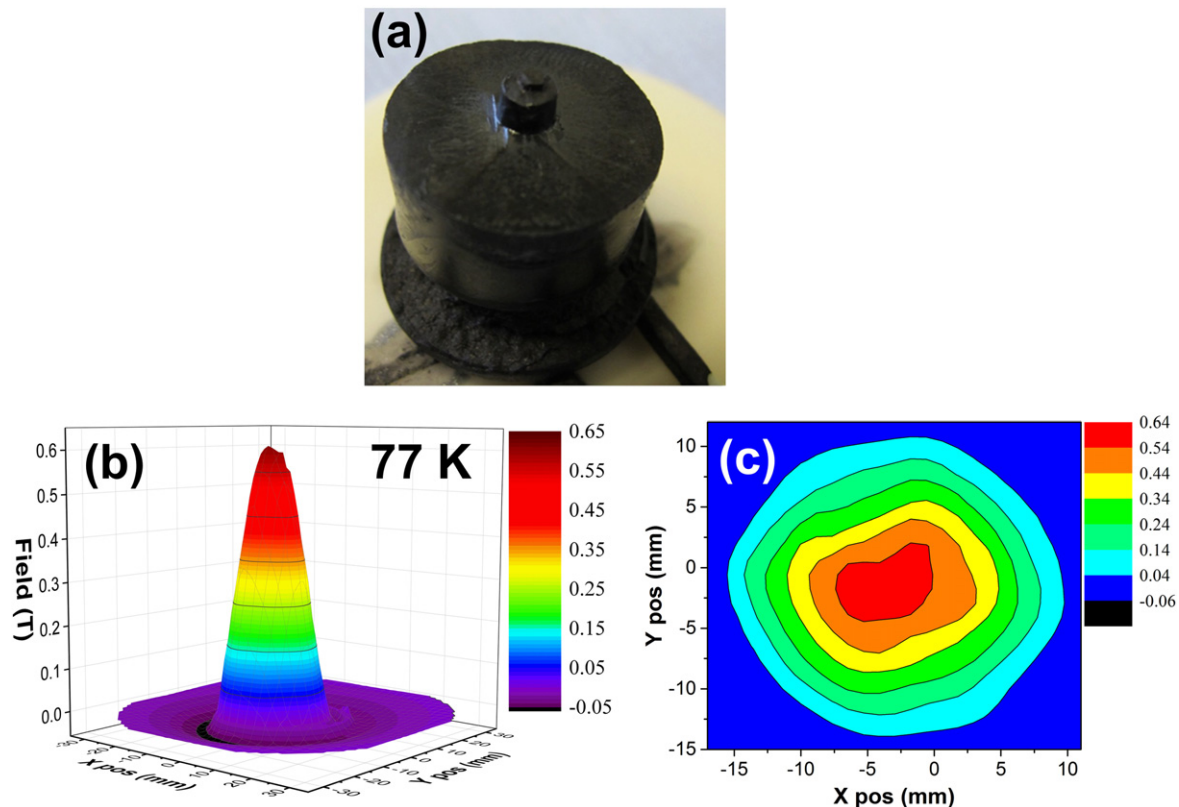
The addition of extra liquid phase constituents to the Y-211 preform prior to SIG resulted in the formation of either voids or regions free of the Y-211 phase in the final microstructure after reaction with Y-211 at the processing temperature employed. Although this pore fraction is small compared to that obtained with conventional TSMG, pores do still remain in the sample microstructure. Hence, from a microstructural aspect, the best configuration appears to be one where the infiltration temperature is controlled and optimised at  $1050\text{ }^{\circ}\text{C}$ . The trapped field profile at 77 K obtained for a YBCO sample fabricated following this approach is shown in figure 9.

A trapped field of 0.69 T at 77 K was measured using a hand-held and scanning Hall probe array on the surface of a single grain YBCO sample processed by BA-SIG. To the best of our knowledge, this is the highest trapped field ever reported by a YBCO sample fabricated by any infiltration and growth process, and is a direct consequence of optimising the Y-211 content in the fully processed sample. There is potential for further fine-tuning of the amount of Y-211 in the single grain composition to improve further the superconducting properties of these technologically important materials.

## 4. Summary and conclusions

SIG is a viable alternative to conventional TSMG since it addresses successfully many of the limitations inherent to the TSMG technique. To date the superconducting properties of SIG processed samples have generally been inferior compared to samples fabricated by TSMG. The reasons for the relatively poor performance of SIG processed samples have been investigated in the present study. The presence of large amounts of Y-211 in the fully processed single grain ( $\sim 41.8\%$ ) is a plausible explanation for this observation. As a result, two new approaches have been investigated in an attempt to control, and subsequently optimise, the Y-211 content in SIG bulk microstructure. Attempts were made to vary the infiltration temperature to enable better control of liquid phase entry into the Y-211 preform during SIG processing and to add additional liquid phase to the Y-211 preform prior to processing. Both these approaches enabled the amount of Y-211 content in the fully processed single grain to be optimised to varying degrees. Control of the liquid phase entry into the Y-211 preform by the use of an appropriate infiltration temperature, from a microstructural aspect, appears to be a more effective approach for tuning the Y-211 content in the single grain because it avoids the formation of large pores in the final sample microstructure. A 25 mm diameter BA-SIG processed YBCO sample, with an optimised Y-211 content of  $\sim 31\%$ , exhibited trapped field of 0.69 T at 77 K. By tuning the Y-211 content, single grain samples processed by SIG, both approaches yield improved superconducting properties and offer potential for fine-tuning the Y-211 content to improve flux pinning further.





**Figure 9.** (a) Photograph of the BA-SIG processed YBCO sample obtained after adjusting the infiltration temperature  $T_i$  to 1050 °C such that a sufficient amount of liquid phase enters the Y-211 preform during processing. Trapped field profiles obtained at a height of 0.8 mm above the YBCO sample surface at 77 K are shown in (b) and (c).

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