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## Single domain YBCO/Ag bulk superconductors fabricated by seeded infiltration and growth

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**Abstract.** We have applied the seeded infiltration and growth (IG) technique to the processing of samples containing Ag in an attempt to fabricate Ag-doped Y-Ba-Cu-O (YBCO) bulk superconductors with enhanced mechanical properties. The IG technique has been used successfully to grow bulk Ag-doped YBCO superconductors of up to 25 mm in diameter in the form of single grains. The distribution of Ag in the parent Y-123 matrix fabricated by the IG technique is observed to be at least as uniform as that in samples grown by conventional top seeded melt growth (TSMG). Fine Y-211 particles were observed to be embedded within the Y-123 matrix for the IG processed samples, leading to a high critical current density,  $J_c$ , of over 70 kA/cm<sup>2</sup> at 77.3 K in self-field. The distribution of Y-211 in the IG sample microstructure, however, is inhomogeneous, which leads to a variation in the spatial distribution of  $J_c$  throughout the bulk matrix. A maximum-trapped field of around 0.43 T at 1.2 mm above the sample surface (i.e. including 0.7 mm for the sensor mould thickness) is observed at liquid nitrogen temperature, despite the relatively small grain size of the sample (20 mm diameter x 7 mm thickness).

### 1. Introduction

The ability of bulk RE-Ba-Cu-O (RE)BCO superconductors to trap magnetic field, and hence their ability to levitate a permanent magnet, is limited severely by mechanical strength, rather than by their superconducting properties. Indeed, these materials are characterized by a relatively low tensile strength of around 10~30 MPa, and fracture frequently when an applied electro-magnetic force exceeds this value [1]. Fabrication of bulk (RE)BCO superconductors with low porosity, therefore, is an important general processing aim for enhancing of the mechanical strength of these material. It has been reported that the tensile strength of void-free Ag-doped SmBCO is 1.3 times greater than that of conventional Ag-doped SmBCO processed by top seeded melt growth (TSMG) [2], demonstrating clearly the correlation of porosity with limited mechanical performance in this material. A relatively

low porosity of around 1 % of the bulk volume fraction has been achieved in samples fabricated by the infiltration and growth technique (IG) compared with samples grown by conventional melt-processes [3]. However, further improvement of the mechanical properties of bulk material is essential if they are to be used in practical applications. The addition of silver to bulk precursor pellets, which strengthens significantly the final melt processed (RE)BCO bulk samples, has been employed commonly in previous studies [4].

This paper reports the use of a seeded IG technique and the silver addition to the precursor powder to improve further the mechanical strength of YBCO bulk superconductors. A study of the microstructure and superconducting properties of samples fabricated by this technique is described.

## 2. Experimental procedure

A sample arrangement consisting of a Y-211 pre-form, a liquid source pellet and a liquid support thin plate was placed on an YSZ bar in a vertical tube furnace. A SmBCO melt textured pseudo single crystal seed of dimensions 3 mm x 3 mm x 2 mm was placed on the top surface of the Y-211 green compact prior to heat treatment (i.e. the so-called cold-seeding method). The final composition of the Ag-doped YBCO bulk, which contained 23 % volume fraction of Y-211 in the Y-123 matrix, was controlled by adjusting the weight ratio of the Y-211 green compact and the liquid source pellet. 0.5 wt.% of Pt was added to the Y-211 precursor powder in order to suppress Y-211 particle ripening during the solidification process. Two types of sample (1 and 2) were prepared for this investigation. Sample 1 (from precursor arrangement 1) was prepared using the Y-211 pre-form and a liquid source pellet containing Ag<sub>2</sub>O. Sample 2 (from precursor arrangement 2) was prepared in the same way as sample 1, but with 50 % of the total amount of Ag<sub>2</sub>O added to the Y-211 and the remaining 50 % of Ag<sub>2</sub>O added to the liquid source pellet. The Ag<sub>2</sub>O content in the all samples was varied from 7.5 wt.% to 20 wt.%, relative to the total weight of the precursor powder. Each powder mixture was pressed uniaxially into a pellet of 20 mm in diameter. A corresponding liquid source pellet of 25 mm in diameter was prepared from a mixture of Y-123, Ba<sub>3</sub>Cu<sub>5</sub>O<sub>8</sub> and Ag<sub>2</sub>O. Raw Y<sub>2</sub>O<sub>3</sub> powder was pressed into a plate of thickness 2 mm and used to support the liquid. Further details of the precursor arrangement can be found elsewhere [3].

The sample was heated to 960 °C at a rate of 200 °C/h, ramped further to 1005 °C, held at this temperature for 2 hours, cooled subsequently to the monotecto-peritectic temperature of 972 °C at a rate of 15 °C/h, cooled slowly to 962 °C at a rate of 0.2 °C/h, then more slowly at a rate of 0.1 °C/h, and finally furnace cooled to room temperature. Note that this temperature profile has been optimised for the fabrication of large, single grain Ag-doped YBCO in previous study [5].

The as-grown samples were oxygenated at temperatures between 550 °C and 400 °C. Half of the fully seeded IG sample was encapsulated in epoxy resin and its surface polished for microstructural observation using a polarized light optical microscope. DC magnetization measurements were performed on the rectangular-shaped samples using a commercial SQUID magnetometer to determine the superconducting transition temperature ( $T_c$ ) and critical current density ( $J_c$ ) under magnetic field applied parallel to the sample *c*-axis.

The trapped magnetic field distribution of the sample was measured using a scanning a hall probe sensor positioned 1.2 mm above the sample surface (i.e. including 0.7 mm for the sensor mould thickness) after field-cooling (FC) to 77 K in a magnetic field of 2 T.

## 3. Results and discussion

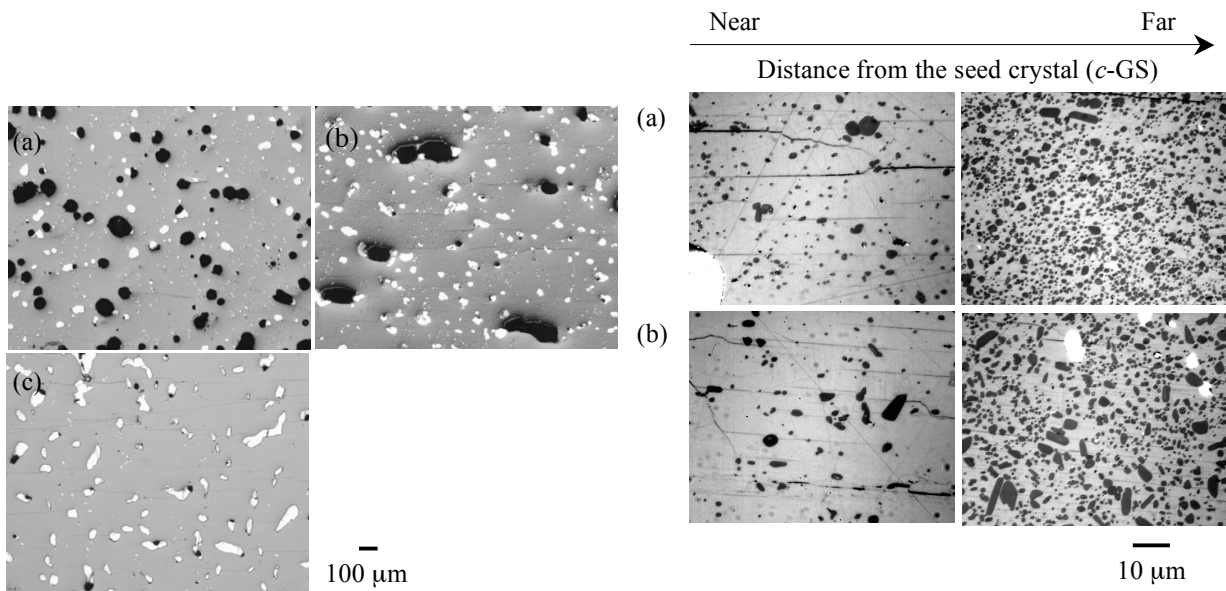
Every melt processed Ag-doped YBCO sample fabricated in this study exhibited a single grain morphology in its top surface. Microstructural observations of the samples prepared from precursor arrangement 1 revealed the presence of an inhomogeneous dispersion of silver in these samples, even though more than 15 wt.% Ag<sub>2</sub>O was added to the Y-211 pre-form. Furthermore, an area of relatively low Ag content observed around the bottom of the pellet is reduced significantly by increasing the Ag<sub>2</sub>O content in the precursor pellet. The distribution of Ag in the Y-123 matrix for the sample containing 12.5 wt.% Ag<sub>2</sub>O, however, is relatively uniform and almost identical to that observed for

the sample containing 10 wt.%  $\text{Ag}_2\text{O}$  fabricated by TSMG. Bulk Ag-doped YBCO samples were fabricated by employing precursor arrangement 2 with a  $\text{Ag}_2\text{O}$  content of 12.5 wt.% based on these results.

Figure 1 shows the sample microstructures for Ag-doped YBCO fabricated by (a) the TSMG technique, (b) the seeded IG technique and (c) the seeded IG technique using precursor arrangement 2 but with metallic Ag added to the Y-211 pre-form rather than  $\text{Ag}_2\text{O}$  (referred to hereafter as the modified precursor arrangement). It is obvious from the micrographs that the pore density of the sample fabricated by TSMG is much greater than that of either of the samples prepared by seeded IG. The surface area fraction of pores,  $S_f$ , for the TSMG grain is around 10%, whereas only 6% porosity is observed for the sample processed by seeded IG using precursor arrangement 2. The density of voids within the final melt processed sample can be further reduced to around  $S_f \sim 2\%$  when metallic Ag powder is added to the Y-211 pre-form rather than  $\text{Ag}_2\text{O}$ , as shown in Fig. 1 (c). A plausible explanation for this is that oxide sources of Ag (e.g.  $\text{Ag}_2\text{O}$ ) decompose to produce oxygen gas during the melt process. This becomes trapped in the sample microstructure on cooling and increases porosity.

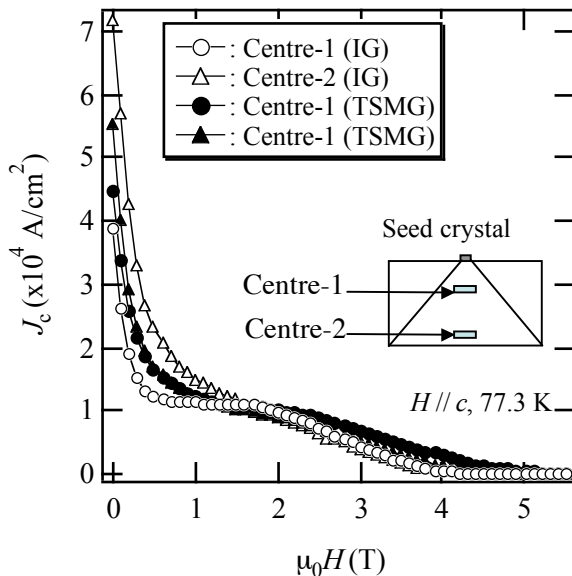
The microstructures under higher magnification of Ag-doped YBCO grains fabricated by seeded IG employing modified precursor arrangement 2 are shown in Fig. 2 (a). The corresponding microstructure for the Ag-doped YBCO sample fabricated by TSMG is shown in Fig. 2 (b) for purposes of comparison. It is clear that no apparent difference in the size and volume fraction of Y-211 particles in the Y-123 matrices around the seed crystal is observed for the samples grown by the seeded IG and TSMG techniques. On the other hand, fine Y-211 particles are observed frequently in the seeded IG microstructure at the limit of  $c$ -growth sector (GS).

All individual small samples cut from parent Ag-doped YBCO fabricated by both the seeded IG technique employing modified precursor arrangement 2 and TSMG exhibit an onset  $T_c$  in excess of 90 K. In addition, the width of the superconducting transition is uniformly less than 2 K in all samples, indicating that the samples are of relatively high quality.

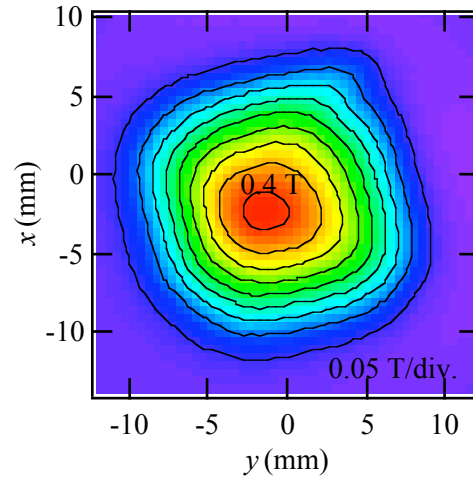


**Figure 1.** Microstructure of Ag-doped YBCO samples grown by (a) TSMG, (b) seeded IG and (c) seeded IG using a modified precursor arrangement incorporating metallic Ag.

**Figure 2.** Optical micrographs in the  $c$ -GS of Ag-doped YBCO samples prepared by (a) seeded IG using a modified precursor arrangement incorporating metallic Ag and (b) TSMG containing 10 wt.%  $\text{Ag}_2\text{O}$ .



**Figure 3.** Field dependence of  $J_c$  at 77.3 K for specimens cut from the  $c$  GS in an Ag-doped YBCO bulk single grain fabricated by seeded IG (open symbols) and TSMG (solid symbols).



**Figure 4.** Contour plot of trapped field of Ag-doped YBCO fabricated by seeded IG employing modified precursor arrangement 2.

Figure 3 shows the field dependence of  $J_c$  for the samples cut from the  $c$  GS in an Ag-doped YBCO bulk single grain fabricated by seeded IG and TSMG techniques. It is clear that the value of  $J_c$  in self-field is observed to increase with increasing distance from the seed crystal for both samples. These results correlate well with the microstructural observations described above. The density of flux pinning centers is small in the vicinity of the seed due to Y-211 particle pushing effects, which leads to a local decrease in  $J_c$ . Furthermore, a clear improvement in  $J_c$  in the lower field region is apparent for the seeded IG samples prepared using modified precursor arrangement 2.

Fig. 4 shows a contour plot of trapped field of Ag-doped YBCO fabricated by seeded IG employing modified precursor arrangement 2. The sample contains no prominent weak-links, which indicates that it has been grown in the form of single grain. A maximum-trapped field of around 0.43 T at 1.2 mm (i.e. including 0.7 mm for the sensor mould thickness) is observed despite the relatively small grain size of 20 mm in diameter.

#### 4. Conclusions

Large single grain of Ag-doped YBCO has been fabricated successfully by a seeded IG technique. A uniform distribution of Ag in the Y-123 matrix has been achieved by adding  $\text{Ag}_2\text{O}$  to both the Y-211 pre-form and the liquid source pellet. The seeded IG sample shows relatively low porosity, whereas a high pore density is observed in samples prepared by TSMG. The density of voids in bulk samples can be decreased further by adding metallic Ag, rather than  $\text{Ag}_2\text{O}$  to the Y-211 pre-form. An inhomogeneous distribution of Y-211 has been observed, leading to a spatial variation of the critical current density within the sample. Finally, an Ag-doped YBCO single grain of 20 mm in diameter fabricated by seeded IG using the modified precursor arrangement incorporating metallic Ag was observed to trap a magnetic field of over 0.4 T at 77 K.

#### Acknowledgments

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