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Effects of densification of precursor pellets on microstructures and critical current properties of YBCO melt-textured bulks

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ABSTRACT

Effects of densification of precursor disks on the density of residual voids and critical current properties for YBCO melt-textured bulk superconductors were systematically investigated. Six YBCO bulks were prepared from precursor pellets with different initial particle sizes of $YBa_2Cu_3O_y$ (Y123) powder and applied pressures for pelletization. It was revealed that use of finer Y123 powder and consolidation using cold-isostatic-pressing (CIP) with higher pressures result in reduction of residual voids at inner regions of bulks and enhance J_c especially under low fields below the second peak.

1. Introduction

RE–Ba–Cu–O (REBCO; RE = rare earth) melt-textured bulk superconductors grown from seed crystals are one of the promising materials for generating strong magnetic fields applicable for various systems, such as desktop NMR [1], magnetic drug delivery [2], and magnetic separation [3]. Trapped field of the superconducting bulk magnet is basically determined by its size, shape and critical current density J_c. So far, various kinds of pinning centers have been introduced to REBCO melt-textured bulks for improving J_c-B characteristics. The interface between superconducting $REBa_2Cu_3O_y$ (RE123) matrix and non-superconducting RE_2BaCuO_5 (RE211) precipitates is one of the representative pinning centers acting under relatively low magnetic fields, and so numerous methods to refine RE211 particles dispersed in RE123 matrix have been studied. For example, addition of Pt is effective in suppression of coarsening of RE211 particles during the melt-growth process [4]. Calcination at moderately low temperatures ~800 °C [5] and ball-milling [6] are effective in reducing the size of starting RE211 powders, resulting in finer RE211 precipitates after the melt-growth process. In contrast, the effects of refinement of starting RE123 powders on J_c properties have been rarely studied. It was reported that the difference in the particle size of starting Y123 powders did not largely affect field-trapping properties at 77 K for YBCO melt-textured bulks [7].

High mechanical strength is also important for realizing bulks with high performance because large electromagnetic force is induced when the bulk traps high magnetic fields generating high density circulating current. The highest trapped field reported to date is 17.6 T at 26 K, which was achieved at the center part of a stack of two Ag-doped GdBCO bulks (25 mmϕ) reinforced by the shrink-fit stainless steel [8].

Dispersed voids are considered as one of the factors deteriorating mechanical and critical current properties of REBCO bulks. Both residual inert gas, such as nitrogen and argon, and oxygen gas generated by the decomposition of RE123 phase at high temperature above ~1000 °C are pointed out as origins of the voids [9]. So far, several effective ways for the elimination and/or reduction of residual voids have been reported. Infiltration Growth (IG) process, in which bulks are grown by infiltration of liquid phases into RE211 preforms, is one of the methods for obtaining REBCO bulks with low porosity [10–12]. For example, the effects of uniaxial compaction pressure applied to the Y211 disks were reported on YBCO bulks grown by IG process [13]. The optimized sample synthesized from the Y211 preform which was pressed under 460 MPa and pre-sintered for 4 h at 950 °C maintained high J_c up to high fields at 77 K (~10^3 A cm^{-2} in 6.5 T) [13]. Besides IG process, addition of Ag, resin impregnation after the heat treatment [14] and melt-growth in pure oxygen atmosphere [15–17] are also effective

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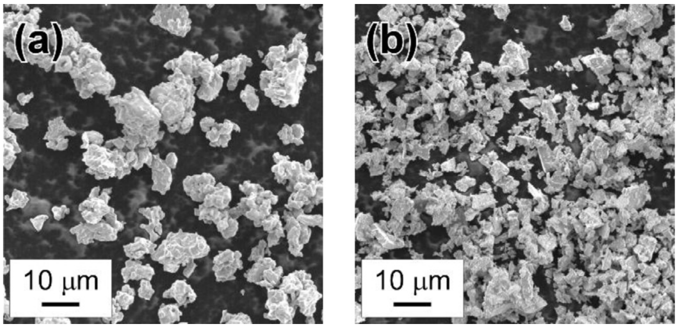

Fig. 1. Secondary electron images for starting powders of Y123-A (a) and Y123-B (b). The mean diameters were 4.58 and 0.71 µm, respectively.

in reducing the voids. However, heat treatment of REBCO bulks at high temperatures in pure oxygen promotes substitution of RE for Ba sites, which degrades the superconducting properties. In our previous study [18], melt-growth of YBCO bulks under pure oxygen atmosphere with a low pressure of ~1 kPa was found to be effective for both reduction of residual voids and suppression of partial substitution of Y for Ba sites. It is also noteworthy that the bulks grown by this method showed largely enhanced J_c in low fields below the second peak field of J_c-B curves. Enhancement of J_c over this field range is substantially important for realizing bulk magnets with high field-trapping properties.

In the present study, effects of densification of precursor disks on the amount of residual voids and critical current properties were investigated for YBCO melt-textured bulks. To prepare the precursor pellets with various packing densities, Y123 powders with different particle sizes were used and they were pressed into a cylindrical shape by applying various pressures using a cold-isostatic-pressing (CIP) apparatus.

2. Experimental

Starting from powder mixtures of Y_2O_3 (99.9% pure), $BaCO_3$ (99.9% pure) and CuO (99.9% pure), two types of Y123 powder materials, Y123-A and Y123-B, were prepared by calcination in air at 880 °C for 24 h and at 925 °C for 50 h, respectively. Y123-B was pulverized by ball-milling after calcination. Fig. 1 shows secondary electron images of these powders. Particle size analyses by the laser diffraction method using SALD-300 V (Shimadzu Corp.) revealed that their mean particle diameters were 4.58 and 0.71 µm, respectively. Y211 powder was also prepared by calcination in air at 800 °C for 120 h. Powders of Y123 and Y211 were mixed into a molar ratio of 7: 3 with addition of Pt powder (0.5 wt%) to suppress the grain growth of Y211. Then the mixture with each 9.0 g in weight was uniaxially pressed into a disk of 20 mmϕ under a pressure of 100 MPa. Some of the disks were additionally pressed by using CIP apparatus (KOBELCO: Dr. CIP) under a pressure of 200 or 300 MPa. After putting a Nd123 single crystal with a typical dimension of ~1 mm × 1 mm × 0.5 mm$^{//c}$ on the center of each precursor pellet with its c-axis vertical to the surface, melt-growth was performed in air by the following temperature pattern. The pellets were heated to 900 °C in 1 h, held at 900 °C for 1 h, heated to 1040 °C in 1 h, held at 1040 °C for 1 h, cooled to 1007 °C in 0.5 h, then slowly cooled to 975 °C at a rate of 0.8 °C/h, and finally cooled in the furnace to room temperature. All the samples listed in Table 1 were successfully grown as bulks with a single domain, as shown in Fig. 2. Rectangular pieces (~2 mm × 2 mm × 1 mm$^{//c}$) were cut from the c-growth regions beneath the seed crystals of the obtained YBCO melt-textured bulks. The distances between the positions of sample pieces and the seed crystal are represented by L hereafter. These small pieces were annealed at 450 °C for 100 h in flowing oxygen. Microstructures were observed by SEM (KEYENCE: VE-7800) and distribution of voids was analyzed using image processing software Image J [19]. Magnetic properties were examined by a SQUID magnetometer (Quantum Design: MPMS-XL5s) up to 5 T and by a vibrating sample magnetometer (VSM) up to 15 T at the High Field Laboratory for Superconducting Materials, IMR, Tohoku University [20]. J_c values were calculated from the width of magnetization hysteresis loops by using the extended Bean model [21]. Flux creep behaviours were examined by magnetic relaxation measurements at 77 K.

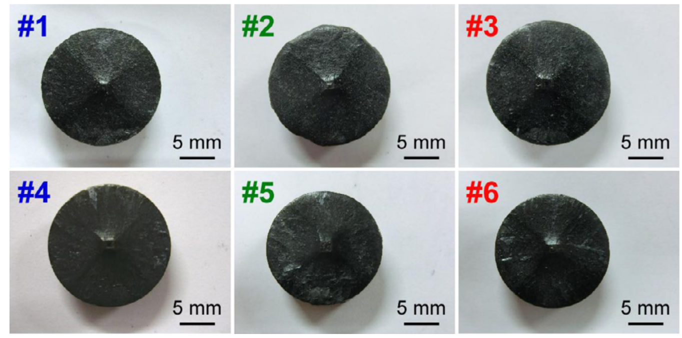

Fig. 2. Top views of YBCO melt-textured bulks #1–6.

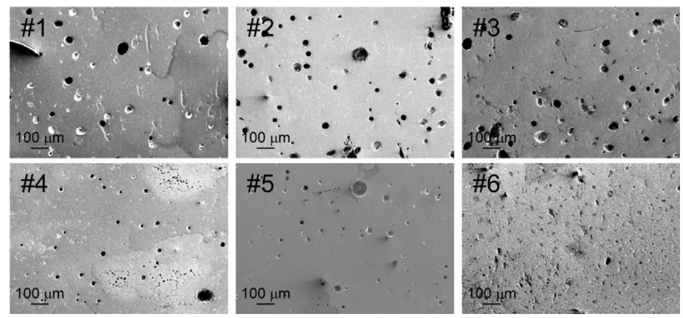

Fig. 3. Secondary electron images for polished *ab*-surfaces at $L = 1$ mm of YBCO melt-textured bulks #1–6.

3. Results and discussion

3.1. Change in sizes and microstructures

Table 1 shows the sizes of precursor pellets and melt-textured bulks prepared by using Y123-A or Y123-B powders and applying pressure by CIP. The volumes of precursor pellets were decreased by applying higher pressure of CIP or use of Y123-B powder, and this tendency was also maintained after the crystal growth.

Microstructural observation revealed that large voids were almost absent in the small specimens cut from the vicinity of the surface ($L = 1$ mm, Fig. 3) for #1–6. This can be explained by relatively easy escape of gas from liquid phases at those regions during melt-growth of Y123. In contrast, a larger number of voids remained in the small pieces cut from $L = 3$ mm of #1–6 as shown in Fig. 4. As expected from the change in the sizes of the bulk samples, the number of large voids was decreased by applying CIP and using fine Y123 powder. As a result of particle imaging analyses, several voids larger than 2×10^4 µm^2 in cross-sectional area were observed in #1 prepared from Y123-A without CIP. On the other hand, such large voids were not observed and more than 70% of voids were smaller than 1×10^3 µm^2 in #4–6 prepared from Y123-B. Area fractions of voids on the polished surfaces were also decreased by using CIP and finer powders as shown in Fig. 4. It was reported that the mechanical properties of REBCO bulks with