Development of homogeneous and high-performance REBCO bulks with various shapes by the single-direction melt growth (SDMG) method

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Abstract
We have developed a single-direction melt growth method in which REBCO melt-textured bulks grow only vertically from a seed plate utilizing the difference in peritectic temperatures of REBCO. Entirely c-grown YBCO, DyBCO and GdBCO bulks with various sizes and shapes were successfully fabricated with high reproducibility. Disk-shaped bulks showed high trapped fields with almost concentric field distributions, reflecting homogeneous and boundaryless bulk structures. In particular, a YBCO bulk with a 32 mm diameter trapped a high field more than 1 T at 77 K. Furthermore, rectangular and joined hexagonal REBCO bulks were successfully fabricated, showing designed field-trapping distributions reflecting their shapes through well-connected superconducting joints among bulks.

Keywords: REBCO, single-direction melt growth, melt grown bulk, trapped field, superconducting joint

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

1. Introduction
REBa2Cu3Oy (REBCO, RE: rare earth element) textured materials have been energetically developed owing to their high critical current density (\(J_c\)) under magnetic fields up to high temperatures, such as the temperature of liquid nitrogen, 77 K. Among them, REBCO melt-textured bulks have been widely studied especially for strong magnets using their high field-trapping (\(B_T\)) properties as high as \(\sim 17\) T, due to the large persistent current circulating in bulks [1–3]. Both the uniformity and reproducibility of \(B_T\) distributions as well as high \(B_T\) of REBCO bulks are essential for extensive applications. Furthermore, bulks with various shapes such as rectangular, hexagonal, fan-shaped, and hollow cylindrical shapes, in addition to typical disk shapes, are required for applications such as desktop NMR/MRI, bearings, undulator and motor systems [4–10]. In order to achieve biaxial texturing, melt growth using a small seed crystal placed on top of the precursor REBCO pellet has been generally adopted, which is well known as ‘top-seeded melt-growth’ (TSMG) or ‘top-seeded infiltration growth’ (TSIG) method. However, it is difficult to fabricate REBCO bulks with \(B_T\) distributions clearly reflecting bulk shapes since the top-seeded bulks are composed of \(a\)-growth and \(c\)-growth regions showing different crystallinity and critical current properties [11, 12]. In addition, fabrication of large bulks requires a very long time for both the crystal growth and oxygenation process, and random nucleation from the

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periphery part of the bulks easily occurs as a result of the large supercooling degree. To address these issues, several attempts have been reported, such as the insertion of a buffer pellet, the addition of BaO$_2$ to decrease both the RE/Ba substitution and the peritectic temperature of REBCO, the modification of seeding, melt growth and oxygenation profiles, control of particle shapes and sizes of precursor powder, multi-seeding and intentional compositional gradient of elements along the radial direction of the bulk [13–21]. Regarding these problems, we have developed an innovative alternative to the top-seeded method, the ‘single-direction melt growth (SDMG)’ method [22]. In this method, large bulk plates cut from commercial TSMG REBCO bulks with relatively high peritectic temperature (T$_p$), such as EuBCO and GdBCO, are used as seed plates. REBCO bulks with T$_p$ lower than that of the seed plate are grown only along a vertical direction from the seed plate. Therefore, grown bulks consist of only a single grain region, and the crystal growth time does not depend on the radial size and shape of the bulks in principle. It is worth mentioning that seed plates can be reused many times after surface polishing, which is one of the advantages of high reproducibility. In the previous report, we succeeded in the fabrication of entirely c-grown YBCO bulks using the SDMG method, which showed better concentrically cone-shaped By distributions compared to those of TSMG processed bulks [22]. In the present study, we have attempted to fabricate entirely c-grown REBCO bulks with various RE elements, sizes and shapes using the SDMG method.

2. Experimental

An overview of the SDMG process is summarized in figure 1. Randomly oriented (RE’ )BCO sintered bulks placed on a large seed plate cut from commercial TSMG (RE’’ )BCO bulks with (001) surface are epitaxially melt grown from the interface to the top, where (RE’’ )BCO must have a T$_p$ higher than that of (RE’ )BCO. In other words, Y or heavy rare earth elements and light rare earth elements are suitable for RE’ and RE’’, respectively. Hereafter, when it is necessary to avoid confusion, the notations RE’ and RE’’ will be used to distinguish RE of grown bulks and seed plates, respectively.

We selected RE’ = Y, Dy, Gd and RE’’ = Gd, Eu, respectively. Precursor powders with a mixture of RE’BCO and RE’$_2$BaCuO$_3$ (RE’211) with a molar ratio of 7:3 for RE’ = Y, Dy and 7:5:2.5 for RE’ = Gd, respectively, were produced by TEP Co. These molar ratios were determined by taking into account the spontaneous partial substitution of RE’ for the Ba site, RE’/Ba substitution, in the RE’BCO crystal. In other words, RE’211 in the starting composition was reduced for bulks with RE’ = Gd, where Gd/Ba substitution proceeds more easily than RE’ = Y and Dy. About 10 wt% Ag$_2$O was mixed with the precursor to decrease T$_p$ and to enhance the mechanical strength of the grown bulks. About 0.5 wt% CeO$_2$ was also added to suppress the grain growth of RE’211 [23, 24]. The precursor powder was pressed into disks with various diameters or other shapes under a uniaxial pressure of ~100 MPa. Precursor RE’BCO bulks were pre-melted for densification, followed by surface-polishing. The pre-melting conditions were as follows; RE’BCO pellets placed on an Al$_2$O$_3$ plate were heat-treated in air at 1015 °C, 1035 °C and 1055 °C for ~2 h for YBCO, DyBCO and GdBCO, respectively, with ramping and cooling rates of ~5 °C min$^{-1}$. The densified RE’BCO bulks with relative density of ~90% were placed on a (001) RE’BCO seed plate with 2–3 mm in thickness cut from commercial TSMG bulks (Gd-QMG and Eu-QMG) with a ~60 mm diameter made by Nippon Steel Co. A slurry consisting of ethanol and precursor powder was coated as a buffer layer at the interface between the bulks and the seed plate to ensure uniform contact. SDMG was carried out in air inside a standard box furnace without intentional temperature gradient. It is important to achieve the partially melting state of the RE’BCO bulk and to avoid deformations of the RE’BCO seed plate that the maximum holding temperature must be above the T$_p$ of the RE’BCO bulk and be below the T$_p$ of the RE’BCO seed plate. The T$_p$ of Ag-added YBCO, DyBCO, GdBCO and EuBCO are ~980 °C, ~990 °C, ~1010 °C and ~1030 °C, respectively, considering that the addition of Ag lowers the T$_p$ by approximately 20 °C [25]. The optimized temperature profiles for RE’BCO bulks with different RE elements and the setting geometry are shown in figure 2 and table 1. After SDMG, grown bulks were separated from the seed plate by cutting using a diamond saw, followed by reductive annealing at 800 °C–850 °C under flowing 1%O$_2$ (Ar balanced) gas for 24 h to suppress the level of the RE/Ba substitution [26]. Finally, oxygenation in a tube furnace was carried out down to 450 °C, 425 °C and 350 °C for YBCO, DyBCO and GdBCO, respectively, for more than 200 h under flowing pure oxygen to achieve the carrier optimally doped state. Therefore, SDMG processed entirely c-grown RE’BCO bulks with various diameters and shapes can be directly obtained on a [001] oriented RE’BCO seed plate. The glossy faces typical of melt-textured bulks can be clearly observed to near the top surface for all of the prepared bulks, suggesting sufficient

![Figure 1. Experimental overview of the single-direction melt growth (SDMG) method.](image-url)
crystal growth of the entire bulks. It is noteworthy that there is almost no failure in crystal growth using the SDMG method under the same temperature profiles indicated in figure 2 and table 1 as long as the placed bulks are in uniform contact with the seed plate, representing high reproducibility of the SDMG method. For comparison, two DyBCO bulks were prepared under the identical condition by the typical TSMG method using a NdBCO single crystal as a seed crystal. Detailed conditions of TSMG are described in our previous paper [27].

In addition to standard disk-shaped bulks, we have demonstrated direct preparation of rectangular and hexagonal-shaped bulks using the SDMG method, where metallic dies with a side length of 16 and 9.2 mm were used for pelletization of the rectangular and the hexagonal bulks, respectively.

For a SDMG YBCO bulk grown on a GdBCO seed plate, crystallinity of the seed plate and the grown bulk was evaluated by the x-ray diffraction (XRD, PANalytical X’PERT PRO MRD) and the bulk/seed interface was observed using a polarized optical microscopy, SEM (ZEISS ULTRA55), and 200 keV transmission electron microscopy (TEM, JEOL JEM-2100) along with elemental analysis using energy dispersive x-ray spectroscopy attached to the SEM and TEM. Small samples for the TEM observation were processed and picked up by the focused ion beam apparatus (Hitachi High-Tech MI4050).

$B_T$ distributions $\sim$0.5 mm above the polished surface of the bulk (seed plate side) were examined by scanning a Hall probe using an axial type sensor (Lake Shore Cryotronics HGCA-3020) at 77 K, where the scanning was performed using a motorized $XY$ stage (SIGMAKOKI HPS120-60XY) at intervals of 1 mm or 2 mm. Magnetization was carried out by field cooling up to 2 T at 77 K. The $B_{T,max}$ is the highest $B_T$ value measured just above the surface of the bulks.

### 3. Results and discussion

First, crystallinities of a GdBCO seed plate and a grown YBCO bulk were evaluated by XRD. Figure 3 shows the pole figures of GdBCO (103) for the seed plate (a) and YBCO (103) for the polished surface on the seed side of the grown bulk (b), respectively. In both figures, four-fold symmetric sharp peaks were observed. The full width at half maximum values of $\Delta \phi$ averaged over four peaks were $\sim$3.5° and $\sim$3.2° for the GdBCO seed and the grown YBCO, respectively. The crystallinity of the grown YBCO was found to be almost unchanged to that of the GdBCO seed plate.

Secondly, the microstructure of the interface at the SDMG grown YBCO/GdBCO seed plate was observed to evaluate the interfacial cleanliness and the degree of interdiffusion of RE elements across the interface. Figure 4(a) shows a polarized optical microscope image of a wide range of the cross-sectional bulk/seed interface. The interface is clean and indistinguishable without coarse impurities and/or cracks, while there are clear differences in the size and distribution of Ag and voids between the seed plate and bulk region. SEM and TEM observations were carried out for the region corresponding to the dashed line in figure 4(a) and the micro area that includes the interface, respectively. Figures 4(b) and (c) show SEM and TEM images around the YBCO/GdBCO interface along with
Figure 3. (103) pole figures of the GdBCO seed plate (a) and the surface on the seed side of the grown YBCO bulk using the SDMG method (b).

Figure 4. Optical (a), SEM (b) and TEM (c) images around the interface between the SDMG YBCO bulk and the GdBCO seed plate. Elemental mappings of Y and Gd corresponding to the area surrounded by the dashed lines are indicated in (b) and (c). Selected area electron diffraction patterns of three different positions across the interface are also shown in (c).

Figure 5. Two-dimensional $B_T$ distributions at 77 K of SDMG processed GdBCO (#S1), DyBCO (#S2 and #S3), YBCO (#S4) and TSMG processed DyBCO (#T1 and #T2) bulks measured ∼0.5 mm above the surface of the bulks. $B_{T,max}$ exhibits the highest $B_T$ value when the Hall probe is in contact with the surface of the bulks.
pellets can be easily realized by the SDMG method through well-connected superconducting joints among the three bulks. It was observed for the S6 joined hexagonal bulk, suggesting little or no decrease in the reflect the shapes of the bulks, indicating the high uniformity plate. It was demonstrated that the appeared using the SDMG method. Figure 6 will not occur in case of the SMDG bulks.

Finally, direct fabrication of bulks with various shapes was attempted using the SDMG method. Figure 6 shows the appearances and B_T distributions of a rectangular YBCO bulk (S5) and a joined hexagonal DyBCO bulk (S6), respectively. The S6 DyBCO bulk was grown from three hexagonal pellets just placed adjacent to each other on a EuBCO seed plate. It was demonstrated that the B_T distributions clearly reflect the shapes of the bulks, indicating the high uniformity in the ab plane of these complex-shaped bulks. Furthermore, little or no decrease in B_T at the boundary of the original pellets was observed for the S6 joined hexagonal bulk, suggesting that well-connected superconducting joints among the three pellets can be easily realized by the SDMG method through simply placing the pellets in contact with each other. Further investigation on the formation of superconducting joints through SDMG is currently underway. In addition, we have succeeded in the direct growth of hollow-cylindrical bulks, for which a few studies have been reported using the TSMG or TSIG method [30, 31], and these results will be reported in a future paper.

In order to evaluate the degree of roundness of B_T distributions for the prepared SDMG and TSMG bulks quantitatively, we adopted the circularity, c, and ellipticity, e, as a scale for comparison of homogeneity. c and e were defined as follows: 
\[ c = 4\pi S/L^2 \]
\[ e = 1 - b/a \]
where S, L, a, and b are area, circumference, semi-major and semi-minor axes of the shapes enclosed by the isomagnetic field lines, respectively. To calculate \( e \), an elliptic approximation of the isomagnetic field lines is applied. c and e are indices that more intensively reflect how disturbed and less symmetric the trapped field distributions are compared to a perfect circle, respectively. Specifically, when the \( c \) and \( e \) are higher, the trapped field distributions are more circular and the ellipticity is lower.

Table 2 summarizes the grown REBCO, preparation method, bulk shape, size, \( B_{T,max} \), circularity, and ellipticity for the prepared bulks in this study. circular and ellipticity, regardless of the RE elements.

| No. | REBCO | Method | Shape       | Size (mm) | \( B_{T,max} \) (77 K) (T) | Circularity, c | Ellipticity, e |
|-----|-------|--------|-------------|-----------|-----------------------------|---------------|---------------|
| #S1 | GdBCO | SDMG   | Disk        | 13.4φ × 8.2' | 0.43                        | 0.983         | 0.050         |
| #S2 | DyBCO | SDMG   | Disk        | 13.6φ × 7.7' | 0.59                        | 0.988         | 0.042         |
| #S3 | YBCO  | SDMG   | Disk        | 20.8φ × 9.9' | 0.79                        | 0.991         | 0.042         |
| #S4 | DyBCO | SDMG   | Disk        | 32.0φ × 12.6' | 1.04                        | 0.965         | 0.100         |
| #T1 | DyBCO | TSMG   | Disk        | 20.0φ × 13.1' | 0.50                        | 0.933         | 0.140         |
| #T2 | DyBCO | TSMG   | Disk        | 20.1φ × 13.2' | 0.47                        | 0.957         | 0.197         |
| #S5 | YBCO  | SDMG   | Rectangle   | 13.1 × 13.1 × 11.3' | 0.57                        | —             | —             |
| #S6 | DyBCO | SDMG   | Joined hexagon | 7.7'(each side) × 10.8' | 0.35                        | —             | —             |

and YBCO bulks with 20.8 and 32.0 mmφ, respectively. These values were comparable with ~0.9 T at 77 K for the reported 24.15 mmφ GdBCO bulk exhibiting the highest B_T properties ever, i.e. B_T >17 T at ~30 K [3]. The quantitative evaluation of the degree of roundness will be discussed later in this paper. Since the SDMG-processed bulks have no acl-growth grain boundaries with homogeneous radial J_c distributions, uniform and concentric B_T distributions are achieved through field-cooling magnetization. Moreover, the B_T-distributions can also be improved for bulks magnetized by the pulsed field method because selective magnetic field invasion through the grain sector region prior to grain boundary region reported in TSMG bulks [28, 29] will not occur in case of the SMDG bulks.

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of the bulks. It should be noted that the reproducibility and roundness of the $B_T$ distributions are slightly low for the large bulks, as seen in $\#4$ YBCO $32.0 \text{ mm} \phi$ bulk. This may be due to the fact that a uniform contact between the seed and the pellet was not sufficiently achieved. More careful surface polishing of the seed plates and optimization of coating conditions of buffer layer will solve this problem.

4. Conclusions

In conclusion, we have developed a ‘SDMG’ method for the fabrication of entirely $c$-grown REBCO melt-textured bulks. In this method, REBCO bulks can be grown directly regardless of bulk diameters and shapes, since bulks are grown only in the vertical direction from a large seed plate cut from commercial REBCO bulks with relatively high peritectic temperatures, such as EuBCO and GdBBCO. Microstructural observation revealed that the bulk/seed interface is clean and that there is very low level of interdiffusion of RE elements, allowing multiple reuses of seed plates with only a little surface polishing. REBCO bulks with various sizes and shapes were successfully grown using the SDMG method for RE = Y, Dy and Gd. Both high circularity and high field trapping properties, i.e. the trapped field value of 1.04 T at 77 K, were achieved for the SDMG bulks. In addition, we have succeeded in the direct growth of bulks with various shapes which exhibited trapped field distributions clearly reflecting their complex shapes including the joined hexagon through achieving well-connected superconducting joints among adjacent multiple bulks. The SDMG method is very promising for the fabrication of homogeneous and high-performance melt-textured bulks with high scalability.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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