Fabrication of high-performance YBa$_2$Cu$_3$O$_y$ melt-textured bulks with selective grain growth

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We developed a simple single-direction melt growth for the fabrication of high-performance REBa$_2$Cu$_3$O$_y$ (RE: rare earth element) melt-textured bulks with selective grain growth. In this method, bulks were epitaxially melt-grown from a large plate cut from commercial melt-textured bulks by utilizing the different peritectic temperatures of REBa$_2$Cu$_3$O$_y$. Ag-added YBa$_2$Cu$_3$O$_y$ bulks with three crystallographic orientations ([100], [110], and [001]) were successfully prepared. The whole c-grown bulk showed a higher trapped field (∼0.39 T) with more circular distributions than the conventional top-seeded bulk. The proposed method is promising for the fabrication of high-performance melt-textured bulks with high scalability and flexibility. © 2020 The Japan Society of Applied Physics

REBa$_2$Cu$_3$O$_y$ (REBCO, RE: rare earth element) tapes and bulks have been energetically developed owing to their high critical current density ($J_c$) under magnetic fields at high temperatures (liquid nitrogen temperature, 77 K). Among them, single-domain REBCO melt-textured bulks have been widely studied for superconducting bulk magnets using their high field-trapping ($B_T$) properties. It is of high importance for REBCO bulks to achieve both an increase in high $B_T$ properties and improvement in the uniformity of $B_T$ distributions for practical applications, such as desktop computer fans, bearings, and motor systems. Since the gradient of the trapped field, $\partial B_T/\partial r$, is proportional to $J_c$, both the enlargement of bulks and improvement in $J_c$ are effective to achieve high $B_T$ max. Many successful efforts have been devoted to enhance $J_c$ through RE-mixing, refinement of RE$_2$BaCuO$_3$ (RE211) particles, introduction of pinning sites, and/or other methods. However, it is difficult to fabricate REBCO melt-textured bulks with homogenous $B_T$ distribution. To achieve the biaxial oriented growth of REBCO, texturization using a seed crystal, such as in top-seeded melt growth (TSMG) or top-seeded infiltration growth (TSIG) methods, has been adopted, resulting in generations of $a$-growth and $c$-growth regions, i.e. grains growing from side surfaces and a bottom surface of a seed crystal, respectively. Pronounced differences in microstructures and $J_c$ properties have been reported between $a$-growth and $c$-growth regions. In general, $c$-growth regions exhibit better crystallinity and higher $J_c$ in magnetic fields with secondary peaks effects than $a$-growth regions which include sub-grain boundaries. On the other hand, the volume fraction of RE211 particles is usually higher in $a$-growth regions, which results in higher $J_c$ in low fields than that of $c$-growth regions. Attempts on the fabrication of REBCO melt-textured bulks mostly composed of $c$-growth regions have been reported by modifying bulk geometry or introducing buffer pellets. In this study, we aimed to fabricate high-performance REBCO melt-textured bulks completely composed of selective grain growth regions (i.e., whole $c$-grown bulks), using a new simple method focusing on the difference of peritectic temperatures ($T_t$) of REBCOs with different rare earth elements, which is referred as the single-direction melt growth (SDMG) method. In this method, randomly oriented sintered (RE)BCO bulks put on a large plate of (RE)BCO melt-textured bulks were epitaxially melt-grown from the interface toward the top, where (RE)BCO must have higher $T_p$ than that of (RE)BCO. In other words, yttrium (Y) or heavy rare earth elements and light rare earth elements are suitable for RE’ and RE”, respectively. In this study, we selected Y and Gd as RE’ and RE”, respectively. Applying this method, we obtained high-performance melt-textured bulks fully composed of single grain growth regions with high scalability. Sintered bulks from the Y–Ba–Cu–O precursor were prepared as follows. Powder mixture consisting of Y$_2$O$_3$ (99.9%), BaCO$_3$ (99.9%), and CuO (99.9%) with a molar ratio of 1.3:1.7:2.4 was calcined twice at $880{\degree}$ C for 12 h to obtain precursor powder composed of YBCO and Y211 with a molar ratio of 7:3. 10 wt% Ag$_2$O was mixed to the precursor to decrease $T_p$ as well as reinforce the mechanical strength of the resulting bulks. In addition, −0.1 wt% Pt was added to suppress the grain growth of Y211. Then, the precursor was uniaxially pressed into disks with a typical diameter of 20 mm. YBCO bulks were sintered in air at $880\degree$ C for 12 h and placed on a seed plate cut from Ag-added GdBCO TSMG bulks (Gd-QMG) provided by Nippon Steel Co., where three crystallographic orientations—[100], [110], and [001]—were investigated for the surface orientations of GdBCO seed plates, as schematically shown in Fig. 1(a). SDMG was carried out in air inside a muffle furnace without using a temperature gradient as in the conventional TSMG and TSIG methods, where the temperature gradient is often applied from the bottom to the top of the sample. The temperature profiles were as follows: samples were heated to $990 \degree$ C for 2 h and held for 1 h, then cooled to $970 \degree$ C for 30 min, slowly cooled to $950 \degree$ C at a rate of 0.5 $\degree$ C h$^{-1}$, and finally furnace-cooled to room temperature. Notably, all the heat treatment processes were carried out below the $T_p$ of Ag-added GdBCO of more than 1000 $\degree$ C and that the melt growth of YBCO was considered to proceed at approximately the $T_p$ of Ag-added YBCO (∼970 $\degree$ C). Prepared bulks were finally oxygenated in a tube furnace from 450 $\degree$ C to 250 $\degree$ C for ∼150 h under oxygen flow after separating the GdBCO seed plates using a diamond saw.
Crystallographic orientations and crystallinity of the prepared bulks were evaluated by means of 0/20 and φ-scan of X-ray diffraction (XRD) measurements (PANalytical, X’Pert Pro, 45 kV, 40 mA). Magnetic susceptibility and magnetization hysteresis loops at 77 K were measured for samples cut from six positions in the whole bulk completely consisting of precipitates extruded from the top surface of the SDMG-[001]-oriented Ag-GdBCO TSMG bulk. To evaluate the position dependence of superconducting properties inside the bulk, the whole bulk with temperature dependences of zero-field cooled (ZFC) magnetization curves at 77 K for samples cut from six positions with an interval of 2 mm in the c-grown Ag-YBCO SDMG bulk. Open and closed symbols represent samples cut along the center and 2 mm from the center, respectively, and the red, blue, and green lines represent samples located at 1, 3, and 5 mm away from the bottom of the bulk, respectively. Temperature dependences of ZFC magnetization are also shown in the inset.

Since the whole c-grown textured bulks can be successfully obtained using the SDMG method, three kinds of Ag-YBCO SDMG bulks were prepared on Ag-GdBCO TSMG bulks with surface orientations [100], [110], and [001], respectively. To evaluate crystallinity, out-of-plane and in-plane XRD measurements were conducted for the polished surfaces of prepared bulks ~1 mm above the seed plate. Figures 3(a) and 3(b) show θ/2θ scans, for example, only 00-20 peaks were observed for the Ag-YBCO bulk grown on the [001]-oriented Ag-GdBCO TSMG bulk, and similarly, the growth directions were confirmed for the other two. In addition, from the in-plane φ scans, only sharp peaks appearing at 90° or 180° intervals were observed, indicating that all three bulks were epitaxially grown using the SDMG method. Therefore, the SDMG method is a promising way to fabricate bulks with selective oriented grain growth. Additionally, we have succeeded in the fabrication of Ag-YBCO SDMG bulks with slightly complex shapes, such as a hollow cylinder and cube, starting from the sintered precursor bulks grown into desired shapes. In the case of these bulks, Bc2 distributions reflecting the shapes of the bulks were clearly observed, which will be reported elsewhere.

Figures 1(a) and 1(b) show schematics of the fabrication of Ag-YBCO SDMG bulks with [100], [110], and [001] orientations using Ag-GdBCO TSMG bulks as seed plates (a), and a photograph of a whole c-grown Ag-YBCO SDMG bulk on the [001]-oriented Ag-GdBCO seed plate (b).
bottom to the top. In addition, almost no interdiffusion between Y and Gd was observed at the interface from the elemental analysis using energy-dispersive X-ray spectroscopy, suggesting that the GdBCO TSMG seed plates can be repeatedly used.

Finally, $B_T$ properties of the whole $c$-grown SDMG bulk were evaluated in comparison to those of the bulk prepared using conventional TSMG method from the same precursor. Figures 4(a)–4(d) show three-dimensional and two-dimensional mappings of the $B_T$ properties at 77 K of the whole $c$-grown Ag-YBCO SDMG bulk (a), (b) and the Ag-YBCO TSMG bulk composed of both $a$-growth and $c$-growth regions (c), (d), respectively. Both bulks exhibited smooth corn-shaped $B_T$ distributions, which indicated that bulks were homogeneous and did not contain any damaged regions. Surprisingly, the whole $c$-grown SDMG bulk exhibited...
higher maximum $B_T$ (~0.39 T) than that of the TSMG bulk (~0.20 T). Moreover, $B_T$ distributions of the SDMG bulk seem to be more circular. To quantitatively evaluate the degree of roundness, we adopted the ellipticity, $e$, and circularity, $c$, as scales for comparison of homogeneity of $B_T$ distributions, where $e$ and $c$ are values that indicate how close the certain ellipse and shape are to the perfect circle, respectively. $e$ and $c$ are defined as follows: $e = 1 - b/a$ and $c = 4\pi S/L^2$, where $a$, $b$, $S$, and $L$ are semimajor and semiminor axes of the ellipse, area and circumference of the shape, respectively. Specifically, when the $B_T$ distribution gets closer to a perfect circle, $e$ approaches to zero while $c$ approaches to one. The averaged values of $e$ and $c$ calculated from the contours of the trapped fields with an interval of 0.02 T shown in Figs. 4(b) and 4(d) are as follows; $e = 0.029$ and 0.061, and $c = 0.991$ and 0.986 for the SDMG and TSMG bulks, respectively. From the quantitative evaluation, the SDMG bulk was decidedly found to show circular $B_T$ distributions. Possible reasons are that horizontal $J_c$ properties inside the SDMG bulk are more position-independent than those of the TSMG bulk because the SDMG bulk is completely composed of c-growth regions.

In conclusion, we developed a new simple SDMG technique for the fabrication of high-performance REBCO melt-textured bulks with selective grain growth. In this method, melt-textured bulks using Y or heavy rare earth elements were successfully melt-grown from the seed plates of Ag-TSMG bulks using light rare earth elements by utilizing the peritectic temperatures of REBCOs with different rare earth elements. Ag-added YBCO bulks with three crystallographic orientations, [100], [110], and [001], were successfully melt-grown from the seed plates of Ag-added GdBCO TSMG bulks. Since the [001]-oriented bulk consists of grains with only c-growth regions, it shows higher trapped fields with more circular distributions than those prepared using the conventional TSMG method. In addition, the bulk grows only vertically using SDMG method. Therefore, the growth time does not depend on the radial size of the bulk, which enables the fabrication of larger bulks in a few days. Thus, the SDMG method is very promising for the fabrication of high-performance melt-textured bulks with high scalability and flexibility.

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