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Top surface morphologies of melt growth processed $Y_{1.5}Ba_2Cu_3O_{7-y}$ bulk superconductors with corner or edge seeding

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ABSTRACT

A corner or edge seeding was attempted to control top surface morphologies (facet lines) of top-seeded melt growth (TSMG) processed $Y_{1.5}Ba_2Cu_3O_{7-y}$ (Y1.5) bulk superconductors. The orientation and numbers of facet lines were successfully modified using the corner/edge seeding with adjusted seed orientations. Most of the facet lines developed on the top surfaces were nearly straight, whereas some of them often had curvatures when the facet lines met the edges with high angles. The size of the growth area of Y123 on the top surfaces was dependent not only on the seeding method but also on the seed orientation. The unreacted regions were often observed on the local parts of the top surfaces, which are attributed to the difference in a growth rate among growth planes. The top surface with the corner seeding where the ⟨110⟩ growth direction is parallel to the diagonal of the Y123 compact showed the highest magnetic flux density and magnetic levitation forces owing to the largest growth area of Y123.

1. Introduction

The top-seeded melt growth (TSMG) process is widely used for the fabrication of single grain $REBa_2Cu_3O_x$ (RE123, RE: rare-earth elements) bulk superconductors with a high magnetic flux density and levitation performance [1–5]. In the conventional TSMG process, a seed is placed at the centre of the top surface of RE123 powder compacts prior to the heat treatment. The seeded RE123 compact is subjected to melt growth heating cycles. When the nucleation of RE123 grains is strictly limited, a RE123 grain grows at the centre seed through a peritectic reaction. During the prolonged isothermal holding just below a peritectic reaction temperature (T_p, 1005 °C) or slow cooling through T_p, only a single RE123 grain grows at the seed without subsidiary RE123 nucleation. Large single grain RE123 bulk superconductors of several cm in diameter can be fabricated using the TSMG process [1,2,5].

The facet lines always developed on the top surface of the single grain RE123 bulk compacts, which is related to the equilibrium shape of RE123 grains in liquid. They begin at the seed and develop along the ⟨110⟩ directions of RE123. The facet lines correspond to specific crystallographic planes in 3-dimensional space, which determine the segregation of RE_2BaCuO_5 (RE211) particles inside the RE123 grains [6–8]. The Y_2BaCuO_5 (Y211) particle segregation in a $YBa_2Cu_3O_x$ (Y123) system is classified into two patterns. One is 1-dimensional linear tracks and the other is 2-dimensional segregation patterns on the polished surfaces. The linear Y211 tracks form in a stoichiometric Y123 system [6], whereas the planar Y211 segregations form in a Y211 excessive Y123 system [8]. In the former case, Y211 particles were trapped along the ⟨110⟩ directions, making *x*-like tracks [6]. In the latter case, Y211 particles are trapped within specific crystallographic planes bounded by the (110) planes and are free in other neighbouring planes, making butterfly-like patterns [7,8]. Since Y211 particles act as flux pinning centres of Y123 [9], the current properties of the regions with more Y211 particles will be higher than those of the regions with less Y211 particles. It is therefore necessary to control the top surface morphologies of TSMG processed RE123 samples to have a uniform Y211 distribution within the Y123 grains. In this study, we attempted corner or edge seeding with controlled seed orientations to modify the growth morphologies of top surfaces of TSMG-processed Y123 bulk superconductors. Based on the top surface morphologies of the TSMG-processed Y123 samples with corner or edge seeding, the magnetic flux density and magnetic levitation forces of the top surfaces are reported.

2. Experimental procedure

The precursor powder used in this study was a mixture of 1 mol Y123 (Solvay Germany, 99.9% purity, 2–3 μm in size) and 0.25 mol Y_2O_3 (BM-CHEM HI-TECH Co., Ltd, China, 99.99% purity, 0.2–3 μm in size) powder, whose nominal composition is $Y_{1.5}Ba_2Cu_3O_{7-y}$ (Y1.5). 1 wt.% CeO_2 powder was added to the powder mixture to refine the Y211 particles [10]. The powder mixture was ball-milled

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for 24 h using ZrO_2 balls. 15 g of the ball-milled Y1.5 powder was uniaxially pressed in a rectangular steel mould (20 mm × 20 mm) into a compact. A Sm123 seed with adjusted seed orientations was placed at the corner or edge of the top surfaces of Y1.5 compacts (see Fig. 1). Tetragons marked by "a" and "b" correspond to corner seeding with orientation relationships of $\langle 100\rangle_{seed}//side_{compact}$ and $\langle 110\rangle_{seed}//side_{compact}$, respectively. Tetragons marked by "c" and "d" correspond to edge seeding with $\langle 110\rangle_{seed}//side_{compact}$ and $\langle 100\rangle_{seed}//side_{compact}$, respectively. From the schematic views, the numbers and orientations of the facet lines that will be developed on the top surfaces can be predicted.

The seeded Y1.5 compacts were subjected to the heating cycles of the melt growth process for the fabrication of single grain Y123 bulk superconductors. The seeded Y1.5 compacts were located at the centre of a box furnace, heated to 1040 °C in air at a heating rate of 100 °C/h held at this temperature for 1 h, cooled to 1020 °C at a rate of 10 °C/h, cooled again to 970 °C at a rate of 0.3 °C/h and finally cooled down to room temperature at a rate of 100 °C/h. For oxygenation, the TSMG processed Y1.5 samples were heated to 500 °C/h at a rate of 100 °C/h in flowing oxygen, held at this temperature for 50 h, cooled to 450 °C at a rate of 100 °C/h, held at this temperature for 100 h, and then cooled to room temperature at a rate of 200 °C/h.

The TSMG processed Y1.5 samples were field-cooled to 77 K using a Nd–B–Fe permanent magnet with a surface field of 0.52 Tesla (T), and the magnetic flux density distribution at 77 K was measured for the top surfaces using a Hall probe. Force–distance (*F*–*d*) curves were measured for field-cooled or zero field-cooled samples to 77 K using a Nd–B–Fe permanent magnet with a diameter of 30 mm and surface magnetic field of 0.5 T. The magnetic levitation forces at 77 K were estimated from *F*–*d* curves.

3. Results and discussion

Fig. 2(a)–(d) shows the top surface views of TSMG processed Y1.5 samples with corner seeding (a and b) and edge seeding (c and d). After TSMG processing, the size of Y1.5 compacts was reduced from 20 mm to 15 mm owing to the volume contraction. It can be seen in all samples that the facet lines are developed on the top surfaces and the shapes are almost similar to those predicted in the schematic view of Fig. 1. One diagonal facet line, which began from a seed and grew to the corner of the compact, is observed at the top surface of sample (a). The entire top surface of sample (a) is covered with a grown Y123 grain without unreacted parts owing to the fast growth along the $\langle 110\rangle$ direction. The top surface morphology with one diagonal facet line corresponds to a quarter of the top surface view of the conventional TSMG processed Y123 superconductors with centre seeding [2], and is similar to that observed in the buffer bridge processed Y123 samples [11]. On the other hand, two facet lines with deviation angles of 3–5° from the sides of the compact are developed

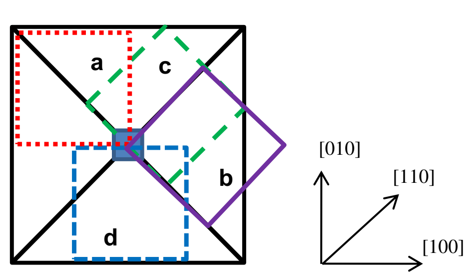

Fig. 1. Schematic view of corner seeding (a and b) and edge seeding (c and d).

(see the lines marked by arrows) on the top surface of sample (b). Unlike the top surface of sample (a), the unreacted region is present at the right upper corner (see the region marked by "r") of the top surface. This is because the growth rate ($R_{G\langle 100\rangle}$) along the $\langle 100\rangle$ direction of Y123 was lower than that along the $\langle 110\rangle$ direction ($R_{G\langle 110\rangle}$). Meanwhile, three facet lines are observed on the top surface of sample (c); one line is almost normal to the side of the compact, and two lines are parallel. Similar to those observed in sample (b), the unreacted regions are also present at the two upper corners of the top surface. On the other hand, two facet lines are observed on the top surfaces of sample (d). One facet line finishes at the corner of the compact and another facet line finishes at the intermediate point of the side. Interestingly, the facet lines have large curvatures, which are comparable to the almost straight facet lines observed in the other samples. The formation of the facet lines with large curvatures appears to be attributed to the unbalanced mass transfer for the growth of Y123 grains in the edge regions.

The top surface morphologies (tracks of facet lines and growth fronts) of Fig. 2 are schematically shown in Fig. 3(a)–(d). The size of the growth area of Y123 on the top surfaces is dependent on the seeding method and seed orientation. The entire top surface was covered with a Y123 grain when the $\langle 110\rangle$ facet line is parallel to the diagonal of the compact (see Fig. 3(a)). The orientation relationship of the corner-seeded Y123 sample is $\langle 100\rangle_{seed}//side_{compact}$. Except sample (a), unreacted regions are observed in the top surfaces of other samples owing to the slower growth rate along $\langle 100\rangle$ directions.

Fig. 4(a) and (b) shows the typical side views (surface morphology) of (a) edge-seeded Y123 and (b) corner seeded Y123 sample after a melt growth process. The side view of sample (a) is divided into a dark isosceles triangle region (c-growth sector) and two upper regions marked by arrows (a/b growth sectors). The c-growth sector is symmetrical in shape because of the equivalent growth of Y123 toward both (left and right) directions from the seed. On the other hand, the c-growth sector of sample (b) is unsymmetrical in shape. It is likely that the Y123 growth to the direction opposite to the corner is easy, whereas the Y123 growth toward the corner is limited.

From the top surface views of Fig. 3 and side views of Fig. 4, the growth nature of Y123 grains in the corner- or edge-seeded Y123 samples was well understood. The two important growing plane/direction families in determining the top surface morphologies are $\{100\}/\langle 100\rangle$ and $\{110\}/\langle 100\rangle$. The growth rate relationship between the two directions is given by

$$R_{G\langle 110\rangle} = \sqrt{2}R_{G\langle 100\rangle} \tag{1}$$

where $R_{G\langle 110\rangle}$ and $R_{G\langle 100\rangle}$ are the growth rate of Y123 along a $\langle 110\rangle$ direction and a $\langle 100\rangle$ direction, respectively. This orientation relationship indicates that the $\langle 110\rangle$ direction should be utilized to obtain a lager Y123 area in the corner or edge seeding.

Fig. 5 shows magnetic flux density (*B*) maps of TSMG processed Y1.5 samples with corner seeding (a and b) or edge seeding (c and d), field-cooled at 77 K using a Nd–B–Fe permanent magnet. The *B* maps of all samples show a single grain flux contour with a single peak near the centre of the maps. No deep valleys or disconnected parts owing to the weakly linked grain boundaries or cracks are observed in all *B* maps. It is pointed out that the *B* maps of samples (a) and (d) are nearly symmetric in shape, whereas the *B* maps of samples (b) and (c) are non-symmetric. The non-symmetric *B* contours of samples (b) and (c) are attributed to the presence of unreacted non-superconducting regions at the upper parts of samples (a) and (d), as already observed in Fig. 2. The maximum *B* values at the peak points of samples (a)–(d) are 2.71 kG, 2.29 kG, 2.24 kG and 1.88 kG, respectively. The results of the magnetic flux density