Effects of RE compositional boundaries on fracture strength at 77 K in large single-grained RE-Ba-Cu-O bulk 150 mm in diameter

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Abstract. Large single-grained RE-Ba-Cu-O high temperature superconducting (HTS) bulk 150 mm in diameter was fabricated by employing the RE compositional gradient technique, where RE denotes rare-earth elements, and fracture strength in the super-large HTS bulk was evaluated at 77 K. No significant difference was observed for the average fracture strength value between the specimens which contained the RE compositional boundaries and the specimens which did not contain the boundaries. The average fracture strength values at 77 K were higher than those at room temperature. These results demonstrate that the fracture strengths of the RE compositional boundaries are not degraded by thermal stress in the cooling process.

1. Introduction

Owing to the excellent and unique features such as large current transport capacity in the presence of strong magnetic fields, automatically stable levitation without active control systems and extremely high trapped field ability in compact space, high temperature superconducting (HTS) bulk materials of RE-Ba-Cu-O (RE: Y or rare-earth elements) are highly attractive for the practical application. Recently, super-large single-grained HTS bulk 150 mm in diameter has been successfully produced (Figure 1) [1]. In order to overcome the problem of undesirable nucleation at the position away from the seed for the super-large HTS bulk, a new process based on the difference in the melting temperature between RE elements, that is, the RE compositional gradient technique was employed in the basic melt-growth processing [1,2].

Since HTS bulks are subjected to electromagnetic force and thermal stress in the superconducting devices [3-5], evaluations of the mechanical properties of HTS bulks are important for the development of superconducting devices equipped with HTS bulks. So far, we have investigated the distribution of the mechanical properties in a super-large HTS bulk 150 mm in diameter through three-point bending tests at room temperature for specimens cut from the bulk [6]. No large difference was observed for the mechanical properties between the specimens which contained the RE compositional...
boundaries and the specimens which did not contain the boundaries [6]. Evaluations of the mechanical properties at cryogenic temperature are informative for the practical application of the super-large HTS bulk. However, the evaluations of the mechanical properties at cryogenic temperature for the super-large HTS bulk have not been carried out extensively [1]. It has not been strictly understood whether the fracture occurs at the RE compositional boundaries or not. In the present study, we investigate the effect of the RE compositional boundaries on the fracture strength at 77 K.

2. Experimental procedure

Evaluations of the fracture strength were carried out through four-point bending tests at 77 K for specimens cut from a super-large single-grained RE-Ba-Cu-O bulk 150 mm in diameter. Specimens were cut from the super-large bulk as shown in Figure 2. The composition ratios of RE-site elements in the precursor of the super-large bulk are also shown. Gd and Dy were chosen as RE. Dy content in the precursor increases with increase of the distance from the seed in order to decrease the peritectic decomposition temperature and suppress the undesirable nucleation at the position away from the seed. The composition ratio of RE-site elements was not changed continuously, but varied step by step, that is, by 5% in the present study. Molar ratio REBa2Cu3Ox:RE2BaCuO5 of the precursor was 3:1. 10 wt% Ag2O and 0.5 wt% Pt were added to the precursor. Crystal growth was conducted by using a (Nd,Sm)-Ba-Cu-O seed in air. Two types of specimens with the dimensions of 2.8 x 2.1 x 24 mm³ were cut from the super-large bulk. Some specimens, denoted as Specimens A1-A5, were cut from the bulk such that the specimens did not contain the RE compositional boundaries between two regions with different Dy content. On the other hand, the other specimens, denoted as Specimens B1-B4, contained the boundaries. These specimens were cut from four layers denoted as 1st-4th layer. Each specimen was placed on the bending test jig and immersed in the liquid nitrogen bath. Four-point bending load was applied as schematically shown in Figure 3 under the crosshead speed of 0.2 mm/min. RE compositional boundary of the Specimens B lies inside the upper loading span where the maximum stress is caused by bending. After the bending tests, RE (Gd and Dy) mapping figures on the 2.1 x 24 mm² side surface of
the fractured specimens were obtained by using micro X-ray fluorescence (µ-XRF) spectrometer.

3. Results and discussion

Fracture strengths of the Specimens A and B evaluated through the four-point bending tests at 77 K are shown in Figure 4. Fracture strengths evaluated at room temperature (RT) are also shown. Specimens for the bending tests at RT were cut from the sites adjacent to the Specimens A1-A3 and B1-B4 of the 2nd and 4th layers. The fracture strength data marked with * was obtained for specimens cut from the 1st layer in our previous study [1]. Few pores were observed for the specimens cut from the 1st layer. On the other hand, the fracture strength data without * was obtained for specimens cut from the 2nd, 3rd and 4th layers in the present study. Porosities of the 2nd, 3rd and 4th layers were 7-14%. The fracture strength values with * are extraordinarily higher than those without *.

Higher fracture strength values with * is attributable to the exceptionally low porosity of the specimens cut from the 1st layer. Such a fracture strength distribution in the super-large HTS bulk is similar to those in smaller HTS bulks [7,8]. Excluding the exceptional data points with *, the average fracture strength values at 77 K of the Specimens A and B were close to each other. The average fracture strength values at RT of the Specimens A and B were also close to each other. These results are similar to the three-point bending test results at RT obtained in the previous study [6]. Although the crystal growth behavior such as growth rate in the super-large HTS bulk can be changed at the RE compositional boundaries, it was found that the boundaries cause no degradation of the fracture strength. In our previous study, no significant differences in the distributions of RE$_2$BaCuO$_5$ particles, Ag particles and pores were observed between the specimens with and without the RE compositional boundaries [6]. Fracture strength values of the super-large HTS bulk at 77 K were higher than those at RT. Such temperature dependence of the fracture strength of the super-large HTS bulk is similar to that of a smaller HTS bulk reported [9]. Improvement of the fracture strength by cooling is consistent with the decrease of inter-atomic distance. These results demonstrate that the fracture strengths of the RE compositional boundaries are not degraded by thermal stress in the cooling process.

No degradation of the fracture strength with increase of the distance from the centre or increase of the Dy content was observed for the super-large HTS bulk as shown in Figure 5.

RE (Gd and Dy) mapping figures on the 2.1 x 24 mm$^2$ side surface of the fractured B1 specimens are shown in Figure 6. Sites where the fracture occurred are marked with arrows. It is observed that the RE compositional boundaries did not always cause the fracture of the specimens, although the fracture of the specimen cut from the 3rd layer occurred at the site adjacent to the boundary. Specimens cut from the 2nd-4th layer contained pores. It is considered that the fracture of these specimens originated from pores or pre-

Figure 4. Fracture strength of the Specimens A and B at 77 K and room temperature. Data points for extraordinarily low porosity specimens cut from 1st layer are marked with *. Average values are obtained excluding data points with *.

Figure 5. Fracture strengths evaluated through four-point bending tests at 77 K for the Specimens A1-A5 and B1-B4 cut from 1st-4th layer.
existing micro-cracks. Since the specimen cut from the 1st layer contained few pores and no significant difference in the fracture strength was observed between the specimens which contained the RE compositional boundaries and the specimens which did not contain the boundaries, it is deduced that the inherent fracture strengths of the RE compositional boundaries and those of non-RE compositional boundary sites are close to each other. While the fractures of the specimens cut from the 1st, 3rd and 4th layers occurred in the Dy rich region, the fracture of the specimen cut from the 2nd layer occurred in the Gd rich region. Although the number of the specimens fractured in the Dy rich region is larger than that in the Gd rich region among the B1 specimens, no degradation of the fracture strength with increase of the Dy content was observed as mentioned above (Figure 5).

4. Summary
Single-grained RE-Ba-Cu-O HTS bulk 150 mm in diameter was fabricated by employing the RE compositional gradient technique. Fracture strength in the super-large HTS bulk was evaluated at 77 K through four-point bending tests for specimens cut from the bulk. Two types of specimens were cut from the bulk: one type was across the boundaries between two regions with different RE compositions and the other was not across the boundaries. There was no significant difference between the average fracture strength values at 77 K of the specimens which had the RE compositional boundaries and specimens which did not contain the boundaries. These fracture strength values at 77 K were higher than those at room temperature. RE mapping revealed that the fracture does not always occur at the RE compositional boundaries. These results demonstrate that the fracture strengths of the RE compositional boundaries are not degraded by thermal stress in the cooling process.

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Figure 6. RE (Gd and Dy) mapping figures on the 2.1 mm x 24 mm² side surface of the fractured B1 specimens. Red region represents Gd rich and green region represents Dy rich. Sites where the fracture occurred are marked with arrows. Specimens cut from (a) 1st layer (Fracture strength: 112 MPa), (b) 2nd layer (Fracture strength: 90 MPa), (c) 3rd layer (Fracture strength: 85 MPa) and (d) 4th layer (Fracture strength: 92 MPa), respectively.