Fracture strength properties of (Gd,Y)BaCuO large single-grain bulk at liquid nitrogen temperature

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Abstract. In order to investigate the fracture strength properties of a (Gd,Y)BaCuO large single-grain bulk with 90 mm in diameter, bending tests were carried out at liquid nitrogen temperature 77 K for the specimens cut from the bulk sample. The large single-grain bulk was fabricated by RE compositional gradient technique to overcome the problem of undesirable nucleation apart from the seed crystal. One precursor that consisted of some regions with different Gd and Y contents was used for the fabrication. Bending tests for the plain and the V-notched specimens were carried out to evaluate the fracture strength and fracture toughness, respectively. The fracture strength values of the specimens that included the interface between two regions with different Gd and Y contents were not lower than those of the specimens that did not include the interface, which is similar to the bending test results at room temperature. The fracture strength values at 77 K were higher than those at room temperature.

1. Introduction
It is well-known that REBaCuO superconducting bulk materials, where RE denotes yttrium or lanthanoid element, have to be a single-grain for their excellent superconducting properties. Thus, REBaCuO bulk materials are fabricated by melt-processing using a seed crystal. Enlargement of REBaCuO bulk size is effective in improving the performances for some superconducting devices. However, it is difficult to obtain large single-grain bulk piece without undesirable nucleation apart from the seed crystal. In order to overcome the problem of undesirable nucleation, RE compositional gradient technique has been developed [1,2]. RE compositional gradient technique is based on the difference in the peritectic decomposition temperature of REBaCuO. One precursor that consists of some regions with different RE contents is used for the fabrication. It has been reported that the trapped magnetic field profile of a large single-grain bulk fabricated by RE compositional gradient technique had single peak [2], which is similar to the trapped magnetic field profiles of smaller REBaCuO single-grain bulks fabricated by using a precursor with no compositional gradient.

Since superconducting bulk materials are subjected to electro-magnetic force and thermal stress in the superconducting devices [3,4], evaluations of the fracture strength properties are indispensable for their practical applications. The present authors previously evaluated the fracture strength of a
(Gd,Y)BaCuO large single-grain bulk fabricated by RE compositional gradient technique through bending tests at room temperature; the fracture strength of the large single-grain bulk was comparable to that of a smaller GdBaCuO bulk fabricated by using a precursor with no compositional gradient [5].

In this study, the fracture strength properties were evaluated at liquid nitrogen temperature 77 K through bending tests for the specimens cut from a (Gd,Y)BaCuO large single-grain bulk sample. It is considered that the evaluations at 77 K are informative for the development of superconducting devices equipped with REBaCuO single-grain bulks.

2. Experimental procedure

Bending test specimens were cut from a (Gd,Y)BaCuO single-grain bulk sample with 90 mm in diameter fabricated by Nippon Steel & Sumitomo Metal Corporation using RE compositional gradient technique. As schematically shown in Figure 1, one precursor with 25 mol% RE$_2$BaCuO$_5$ (RE211) was prepared such that the Y content increased with increase of the distance from the centre of it. 1 wt% Ce was added to the precursor to inhibit coarsening of RE211 particles [6,7]. 16.7 wt% Ag was also added to the precursor. Ag addition is effective in improving the fracture strength properties [8-10]. The precursor was heated in air up to 1443 K, kept at that temperature for 1 h and then cooled down to 1310 K. After that, one (Nd,Sm)BaCuO seed crystal was placed on the top of it. Oxygen annealing was conducted at 673 K for 100 h for the melt-grown bulk sample.

Bending test specimens with the dimensions of 2.8 x 2.1 x 24 mm$^3$ were cut from the bulk sample as schematically shown in Figure 1. Specimens that did not include the interface between two regions with different Gd and Y contents are denoted as Specimens A. On the other hand, specimens that included the interface are denoted as Specimens B. Plain specimens and V-notched specimens were used for the evaluations of the fracture strength and fracture toughness, respectively, as schematically shown in Figure 2 (a) and (b). While Specimens A1, A3, A5, B1 and B2 were used for the fracture strength evaluation, Specimens A2, A4 and A6 were used for the fracture toughness evaluation. V-notch was introduced by using razor with diamond paste. The notch root radius was about 20 μm.

![Figure 1. Schematic illustration of cutting out of bending test specimens.](image1)

![Figure 2. Schematic illustration of 4-point bending tests for (a) fracture strength and (b) fracture toughness evaluations, respectively.](image2)
point bending load was applied at 77 K under the crosshead speed of 0.2 mm/min. Before the loading, the specimen was immersed into the liquid nitrogen bath, together with the 4-point bending jig. Fracture strength $\sigma$ was calculated by the following equation.

\[
\sigma = \frac{3P(L - l)}{2wt^2}
\]

Where $P$ is the maximum load, $L$ is outer supporting span, $l$ is inner loading span, $w$ and $t$ are width and thickness of the plain specimens, respectively. Fracture toughness $K_{IC}$ was calculated by the following equations [11].

\[
K_{IC} = \sigma \sqrt{\pi a F(a/W)}
\]

\[
\sigma = \frac{3P(L - l)}{2BW^2}
\]

\[
F(a/W) = \frac{2W}{\pi a} \tan(\frac{\pi a}{2W}) - 0.923 + 0.199(1 - \sin \frac{\pi a}{2W})^4
\]

\[
\cos \frac{\pi a}{2W}
\]

Where $\sigma$ is the fracture strength, $P$ is the maximum load, $a$ is V-notch depth, $L$ is outer supporting span, $l$ is inner loading span, $W$ and $B$ are height and thickness of the V-notched specimens, respectively. Fracture surfaces of the V-notched specimens were observed by using digital microscope KEYENCE VHX-500 with 3D observation mode. The V-notch depth was measured on the fracture surfaces by using image analysis software.

3. Fracture strength at 77 K

Figure 3 presents the fracture strength values at 77 K of Specimens A1, A3, A5, B1 and B2 cut from 1st-4th layers of the bulk sample (see Figure 1). Open and solid symbols represent the data of Specimens A and B, respectively. Since it has been reported that the fracture strength of YBaCuO bulk was superior to those of other REBaCuO bulks [12], it was expected that the fracture strength increased with increase of the distance from the centre of the bulk sample. However, such an increase is not observed for the bending test results. The fracture strength values of Specimens B2 are slightly lower than those of Specimens B1. However, the distributions of pores and RE211 particles of Specimens B1 and B2 were similar to each other.

Figure 4 presents comparison of the fracture strength at 77 K between Specimens A and B. The average fracture strength values at room temperature reported elsewhere [5] are also shown for reference. The fracture strength values of Specimens B are not lower than those of Specimens A. Thus, it is demonstrated that the interfaces between two regions with different Gd and Y contents do not cause the deterioration of the fracture strength for the whole bulk material. The average fracture strength values at 77 K are higher than those at room temperature. It is deduced that such an improvement of the fracture strength is mainly attributable to the decrease of the inter-atomic distance by cooling. It has been reported that the fracture strength values of conventional GdBaCuO single-grain bulks with 30-40 mm in diameter fabricated by using a precursor with no compositional gradient were 97 MPa at 77 K [13], which was obtained under the same testing conditions as those of the bending test carried out in this study, and 69-115 MPa at room temperature [10]. The fracture strength value commonly depends on the testing conditions, such as the dimensions of specimen and loading method. Thus, it is considered that the fracture strength of the (Gd,Y)BaCuO large single-grain bulk fabricated by RE compositional technique is comparable to that of the smaller GdBaCuO single-grain bulk. It has been reported that the fracture strength values at 77 K of a (Gd,Dy)BaCuO bulk fabricated
by RE compositional gradient technique were around 90 MPa [14]. Thus, it is also considered that the fracture strength values of the (Gd,Y)BaCuO bulk tested in this study are comparable to those of the (Gd,Dy)BaCuO bulk.

Figure 5 presents Weibull plots of the fracture strength data at 77 K. F, σ and m represent cumulative fracture probability, fracture strength and Weibull coefficient, respectively. Weibull coefficient values of the fracture strength at room temperature [5] are also shown for reference. Weibull coefficient value is obtained through linear fitting of the Weibull plot data, and it is commonly used for the evaluation of scatter of the data; larger Weibull coefficient value means smaller scatter of the data. Weibull coefficient values of Specimens A are smaller than those of Specimens B. It is deduced that the smaller Weibull coefficient values of Specimens A are associated with the large distance between Specimens A1 and A5. There is no significant difference in the Weibull coefficient value between 77 K and room temperature. Meanwhile, it seems that fitted curve with curvature changing in the middle, which consists of two straight lines, can be drawn for the Weibull plot data, so there is a possibility that this bulk has two fracture modes.

Figure 3. Fracture strength values at 77 K for Specimens A1, A3, A5, B1 and B2 cut from 1st-4th layers of the bulk sample (see Figure 1).

Figure 4. Fracture strength of Specimens A and B at 77 K. The average fracture strength values at room temperature reported elsewhere [5] are also shown for reference.

Figure 5. Weibull plots of the fracture strength data at 77 K. F, σ and m represent cumulative fracture probability, fracture strength and Weibull coefficient, respectively. Weibull coefficient values of fracture strength at room temperature [5] are also shown for reference.
4. Fracture toughness at 77 K
Figure 6 presents the fracture surface of a V-notched specimen. In order to evaluate the fracture toughness, V-notch depth was measured on the fracture surfaces. Figure 7 presents the fracture toughness values at 77 K of Specimens A2, A4 and A6. Since it is impossible to adjust the V-notch to the interface between two regions with different Gd and Y contents, the fracture toughness tests of Specimens B were not carried out. It has been reported that the fracture toughness values at 77 K of a (Gd,Dy)BaCuO large single-grain bulk fabricated by RE compositional gradient technique were around 1.0-1.8 MPa m$^{1/2}$ [15]. Fracture toughness values of the (Gd,Y)BaCuO bulk tested in this study are comparable to the values of the (Gd,Dy)BaCuO. While the fracture strength value does not increase with increase of the distance from the seed crystal as mentioned above, the fracture toughness value slightly increases. It is considered that Y addition is effective in improving the crack propagation resistance.

![Figure 6. Fracture surface of V-notched specimen.](image)

![Figure 7. Fracture toughness of Specimens A2, A4 and A6 evaluated through bending tests at 77 K for V-notched specimens.](image)

5. Summary
In order to investigate the fracture strength properties of a (Gd,Y)BaCuO large single-grain bulk with 90 mm in diameter fabricated by RE compositional gradient technique, bending tests were carried out at 77 K for the plain and the V-notched specimens cut from the bulk sample. The fracture strength values at 77 K of the specimens that included the interface between two regions with different Gd and Y contents were not lower than those of the specimens that did not include the interface, which is similar to the bending test results at room temperature. The fracture strength at 77 K evaluated in this study was superior to that at room temperature evaluated in the previous study. The fracture strength of the large single-grain bulk was comparable to those of smaller single-grain bulks fabricated by using a precursor with no compositional gradient. While the fracture strength values did not increase with increase of the distance from the seed crystal, the fracture toughness values slightly increased.

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