Fracture toughness of low porosity Dy123 bulks melt-processed in pure oxygen

A Murakami\textsuperscript{1}, H Miyata\textsuperscript{1}, R Hashimoto\textsuperscript{1} and K Katagiri\textsuperscript{2}

\textsuperscript{1} Graduate School of Science and Technology, Hirosaki University, 3 Bunkyo-cho, Hirosaki, Aomori 036-8561, Japan
\textsuperscript{2} Faculty of Engineering, Iwate University, 4-3-5 Ueda, Morioka, Iwate 020-8551, Japan
E-mail: amura@mech.hirosaki-u.ac.jp

Abstract. Fracture toughness evaluations of specimens cut from low porosity Dy123 single-grain bulks melt-processed in pure oxygen were carried out at room temperature. While the porosity of a conventional Dy123 single-grain bulk melt-processed in air measured in the previous study is about 20\%, those of the Dy123 bulks melt-processed in pure oxygen are below 4\%. The minimum of the fracture toughness values of the low porosity bulk is 15\% higher than that of the conventional bulk, and the maximum of the former and that of the latter are close to each other. The difference in the fracture toughness among these bulks is discussed in association with the porosity and the mechanical properties of the matrix evaluated by Vickers indentation tests.

1. Introduction

Various devices using R123 (RBa\textsubscript{2}Cu\textsubscript{3}O\textsubscript{x}, where R is yttrium or rare-earth elements) single-grain bulk superconductors, such as quasi-permanent magnets, current leads and current limiters, are requested. Since bulk materials are brittle and have pre-existing micro-cracks, improvement and understanding of the fracture toughness are indispensable for practical applications of them. The present authors previously reported the fracture toughness of Dy123 bulks melt-processed in air; the fracture toughness near the top surface of the bulks is excellent, mainly due to the large net cross-sectional area of the specimen in association with the low porosity [1]. According to this result, the fracture toughness might be improved by decreasing the porosity in the whole region of bulk materials. It has been reported that the porosity of bulk materials can be decreased by increasing the oxygen pressure in the melt-processing [2, 3] and the tensile strength of plain specimens cut from a Sm123 bulk melt-processed in pure oxygen is 30\% higher than that of a bulk melt-processed in 1\% O\textsubscript{2}-Ar atmosphere [2]. There are some reports about the fracture toughness evaluations of bulk materials [1, 4-6]. However, the fracture toughness of low porosity bulk materials melt-processed in high oxygen pressure has not been investigated. In this study, fracture toughness evaluations with SEVNB (single-edge V-notched beam) method were carried out for low porosity Dy123 bulks melt-processed in pure oxygen. Furthermore, characteristics of the fracture toughness of these bulks are discussed in association with the porosity and the mechanical properties of the matrix evaluated by Vickers indentation tests.
2. Experimental

2.1. Preparation of fracture toughness test specimens

Disc-shaped Dy123 single-grain bulk samples fabricated by Nippon Steel Corporation were tested. Figure 1 shows a schematic illustration of melt-processing of the bulk samples. The precursor of the bulk samples were heated in oxygen or air up to 1423 K, kept at this temperature for 1 hour and then cooled down to 1313 K. After that, one Nd123 seed crystal was put on the top of each bulk and the bulks were gradually cooled down in air. Fracture toughness test specimens with $2.8 \times 2.1 \times 24 \text{ mm}^3$ were cut from the bulk samples such that the 2.1 mm direction almost corresponded to the c-axis of the bulk samples. Oxygen anneal was conducted at 723 K for 100 hours for the specimens. Specifications of the specimens are shown in Table 1. The specimens are denoted as ON, OY and AY, respectively.

Figure 2 (a) and (b) show optical micrographs of the polished surface of the specimen OY and the specimen AY, respectively. The porosity of the specimen AY cut from the inner region of the bulk sample is about 20% [1]. On the other hand, few pores are observed for the specimen ON and the specimen OY.

2.2. Fracture toughness test procedures

Figure 3 shows a schematic illustration of SEVNB fracture toughness test. After the oxygen annealing, a notch was introduced at the center in the longitudinal direction of the specimen by using a slow-speed micro-cutter with a diamond blade. The notch root was finished by a razor blade with diamond paste to make the root radius about 20 µm. The notch depth was 0.91-1.19 mm. 3-point bending load was applied at room temperature by means of the INSTRON 4464 testing machine at constant displacement rate of 0.1 mm/min. The fracture toughness value $K_{IC}$ was calculated by the following equations (1) and (2) [7].

$$K_{IC} = \left(\frac{PS}{BW}\right)^\frac{3}{2} \cdot \left(\frac{a}{W}\right)^\frac{1}{2} \cdot Y\left(\frac{a}{W}\right)$$

$$Y\left(\frac{a}{W}\right) = 1.964 - 2.837 \left(\frac{a}{W}\right) + 13.711 \left(\frac{a}{W}\right)^2 - 23.250 \left(\frac{a}{W}\right)^3 + 24.129 \left(\frac{a}{W}\right)^4$$

where $P$ is the maximum load applied, $a$ is the V-notch depth, $S$ is the fulcrum span (21 mm), $W$ and $B$ are the height and the thickness of the specimens, respectively.

Side surfaces of the fractured specimens were polished by using lapping sheets and observed through an optical microscope. The area fraction of pore (porosity) was evaluated on the polished surfaces with 1.6 x 2.4 mm² by using image analysis software. After that, Vickers indentation tests were conducted on the polished surfaces by using AKASHI Mvk Type C at room temperature. The Vickers hardness $HV$ and the fracture toughness $K_c$ evaluated by IF (indentation fracture) method were calculated by the following equations (3) and (4) [7].

$$HV = 1.8544 \frac{Q}{(2d)^2}$$

$$K_c = 0.018 \left(\frac{E}{HV}\right)^\frac{1}{2} \frac{Q}{C^\frac{1}{2}} = 0.026 \frac{E^\frac{1}{2}Q^\frac{1}{2}d}{C^\frac{3}{2}}$$

where $Q$ is the indentation load applied (4.9 N), $d$ is a half of the average length of the diagonals of the indentation scar, $E$ is the Young’s modulus, $C$ is a half of the average length between the tip of cracks initiated from corners of the indentation scar, respectively. The cracks were measured after 30 seconds.
**Figure 1.** Schematic illustration of melt-processing.

**Table 1.** Specifications of fracture toughness test specimens cut from Dy123 bulk samples.

|        | Dy211 [mol%] | Pt [wt.%] | Atmosphere in melt-processing | Oxygen anneal | The number of specimens | Diameter x thickness of bulk samples [mm x mm] |
|--------|--------------|-----------|--------------------------------|---------------|-------------------------|-----------------------------------------------|
| ON     | 25           | 0.5       | Oxygen                         | No            | 3                       | 45 x 25                                       |
| OY     | 25           | 0.5       | Oxygen                         | Yes           | 4                       | 65 x 14                                       |
| AY     | 25           | 0.5       | Air                            | Yes           | 7                       | 30 x 10                                       |

**Figure 2.** Optical micrographs of (a) specimen OY and (b) specimen AY.

**Figure 3.** Schematic illustration of SEVNB fracture toughness test.
3. Results and discussion

3.1. SEVNB fracture toughness test

Figure 4 shows fracture toughness values of the specimen ON and the specimen OY, together with the minimum, average and maximum values of the specimen AY [1]. The fracture toughness values of the specimen ON scatter from 1.06 to 1.11 MPa m$^{1/2}$, and those of the specimen OY scatter from 1.39 to 1.63 MPa m$^{1/2}$. These averages are 1.09 and 1.50 MPa m$^{1/2}$, respectively. It is thought that the fracture toughness is improved by oxygen annealing which is indispensable for the excellent superconducting properties of bulk materials.

The minimum of the fracture toughness values of the specimen OY is 15 % higher than that of the specimen AY, and the maximum of the former and that of the latter are close to each other. The maximum value of the specimen AY is obtained for a specimen with an extraordinarily low porosity cut from the region near the top surface of the bulk sample. This improvement of the fracture toughness by decreasing the porosity is similar to the improvement of the tensile strength evaluated by using plain specimens cut from Sm123 bulks [2].

Figure 5 shows the relationship between the fracture toughness value and the porosity of the specimen ON and the specimen OY. A broken line indicates the extrapolation of data of the specimen AY reported elsewhere; the fracture toughness value at 0 % porosity was estimated to be 1.75 MPa m$^{1/2}$ [1]. There is no significant difference in the porosity between the specimen ON and the specimen OY, which are below 4 %. Thus, it is deduced that the improvement of the fracture toughness by oxygen annealing is ascribed to the phase transformation from tetragonal to orthorhombic in the oxygen annealing, pre-existing micro-cracks perpendicular to the crack propagation direction induced in the oxygen annealing and so on. The fracture toughness values of the specimen OY are slightly lower than the values estimated by the extrapolation of data of the specimen AY.

3.2. Vickers indentation test

In order to investigate the mechanical properties of the matrix of the specimens, Vickers indentation tests were carried out. Figure 6 (a) and (b) show optical micrographs of the Vickers indentation scar of the specimen ON and the specimen OY by indenting in the c-axis. The outline of the scar and cracks initiated from corners of the scar are clearly recognized for the specimen ON. On the other hand, the outline of the scar of the specimen OY is not clear (shown by broken lines in the figure) because delamination of the matrix occurred due to the many pre-existing micro-cracks perpendicular to the c-
Figure 6. Optical micrographs of Vickers indentation scar of (a) specimen ON and (b) specimen OY.

Figure 7. Vickers hardness by indenting in the c-axis ($Q \parallel c$) and perpendicular to it ($Q \perp c$).

Figure 8. Fracture toughness evaluated by IF method with indenting in the c-axis.

axis induced by oxygen annealing. Cracks initiated from the scar are not also clear. The scar of the specimen AY that also includes the many pre-existing micro-cracks is similar to that of the specimen OY.

The Vickers hardness values are shown in Figure 7. Vickers indentation tests were conducted on the polished side surfaces of the fractured specimens as mentioned above. Although it is suspected that the hardness values depend on the distance from the fracture surface, such a dependency is not observed.

The hardness of the specimen AY by indenting in the c-axis and that in the direction perpendicular to it are almost equal to each other. This is consistent with the hardness characteristics of an Y123 bulk [8]. On the other hand, the hardness of the specimen ON, which is higher than that of the specimen OY, shows significant anisotropy in comparison with those of the other annealed specimens OY and AY. It is deduced that this inconsistency is ascribed to the phase transformation in the oxygen annealing and the behavior of the many pre-existing micro-cracks perpendicular to the c-axis in the annealed specimens by indenting in the c-axis; pressing and wedging. The hardness of the specimen AY is higher than that of the specimen OY. It is thought that the mechanical properties of the matrix depend on the oxygen pressure in the melt-processing, the dimensions of the bulk (see Table 1) and so on.

Figure 8 shows the fracture toughness values evaluated by IF method with indenting in the c-axis, together with the average values obtained by SEVNB method mentioned above. Since cracks initiated from the indentation scar of the annealed specimens OY and AY can not be clearly recognized as mentioned above, it is assumed that $C$ corresponds to a half of the average of the $d_1$ and $d_2$ of delaminated region around the scar (see Figure 6 (b)). The averages of the Young’s modulus values evaluated by bending test of the specimens ON and OY [9] and the Young’s modulus value at 0 % porosity estimated by the extrapolation of data of bending test of the specimen AY [10] are used for
the calculation of the fracture toughness value of each specimen; these are 162, 153 and 168 GPa, respectively.

The average value obtained by IF method and that by SEVNB method of the low porosity specimen OY are almost equal to each other. It was expected that the fracture toughness evaluated by IF method was higher than that by SEVNB method for the specimen AY that includes pores. However, the former is lower than the latter, which is consistent with the fracture toughness comparison of Sm123 bulks [6].

The fracture toughness evaluated by IF method for the specimen OY is higher than that for the specimen ON. This coincides with the fracture toughness evaluated by SEVNB method mentioned above. It was also expected that the fracture toughness evaluated by IF method for the specimen OY is slightly lower than that for the specimen AY because the fracture toughness values obtained by SEVNB method for the specimen OY are slightly lower than the values estimated by the extrapolation of data of the specimen AY as mentioned above. However, the former is slightly higher than the latter. The reason for this inconsistency has not been understood yet.

4. Summary
Fracture toughness evaluations of specimens cut from low porosity Dy123 single-grain bulks melt-processed in pure oxygen were carried out at room temperature. While the porosity of a conventional Dy123 single-grain bulk melt-processed in air measured in the previous study is about 20 %, those of the low porosity Dy123 bulks are below 4 %. The minimum of the fracture toughness values of the low porosity bulk is 15 % higher than that of the conventional bulk, and the maximum of the former is close to that of the latter obtained for a specimen with an extraordinarily low porosity cut from the region near the top surface of the bulk. The fracture toughness values of the low porosity bulk are slightly lower than the values estimated by the extrapolation of data of the conventional bulk in the previous study. There is a slight difference in the mechanical properties of the matrix evaluated by Vickers indentation tests between the low porosity bulk and the conventional bulk. Understanding the reason for it will be informative for further improvement of the mechanical properties of the low porosity bulks.

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