Effects of Ag particle and pore distributions on fracture toughness of Dy123 bulks

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Abstract. Fracture toughness evaluations on Dy123 single-grain bulks with X wt.% Ag (X=5, 7.5 and 10) melt-processed in air were carried out at room temperature, and the scattering of the fracture toughness values is discussed in terms of the distributions of pore and Ag particle in these bulks. The fracture toughness is improved by the Ag addition; the average values are 1.34, 1.44 and 1.54 MPa m¹/² for X=5, 7.5 and 10, respectively. The porosity near the top surface of these bulks is distinctly low. In X=5, an increase of the fracture toughness value with decrease of the porosity is clearly observed. According to the extrapolation of data, it is deduced that the fracture toughness value at 0% porosity will be 20% higher than the average value.

1. Introduction
Practical applications of R123 (RBa₂Cu₃Oₓ, where R is yttrium or rare-earth elements) single-grain bulk superconductors are partly limited by their mechanical properties. Since the bulk materials have pre-existing micro-cracks besides the intrinsic brittleness, understanding and improvement of the fracture toughness are indispensable for practical applications of them.

Since Dy123 bulks have low thermal conductivities [1] besides the excellent superconducting properties [2], they are expected to be applied to bulk current leads [3]. Although it is well-known that Ag addition is effective in improving the mechanical properties of the bulk materials [4-9], increase of the thermal conductivity is inevitable. Then, investigation about the mechanical properties of Dy123 bulks with small amount of Ag additives will be useful for development and practical application of Dy123 bulk current leads. However, the mechanical properties of the bulk materials with Ag additives less than 10 wt.% have not been investigated extensively [9]. Furthermore, relationship between the mechanical properties and the distributions of pore and Ag particle in the bulk materials has not been understood enough. The present authors recently reported the mechanical properties of Dy123 bulks with 5, 7.5 and 10 wt.% Ag evaluated by bending test of plain specimens; the bending strength increases and the Young’s modulus decreases with increase of the Ag content [9]. The fracture toughness of Dy123 bulks without Ag has been also reported; the fracture toughness increases with decrease of the porosity [10].

In this study, fracture toughness evaluations on Dy123 single-grain bulks with 5, 7.5 and 10 wt.% Ag melt-processed in air were carried out at room temperature, and the correlation between the present
results and the mechanical properties of various Dy123 bulks obtained in the previous studies is discussed in terms of the distributions of pore and Ag particle in these bulks.

2. Experimental

Dy123 single-grain bulk samples with X wt.% Ag (X=5, 7.5 and 10), 25 mol% Dy211 (Dy2BaCuO5) and 0.5 wt.% Pt fabricated by Nippon Steel Corporation were tested. The diameter and the thickness of these bulk samples are 46 and 25 mm, respectively. Precursors of these bulk samples were heated in air up to 1423 K, kept for 1 hour and then cooled down to 1313 K. After that, one Nd123 seed crystal was placed on the top of each bulk and the bulks were gradually cooled down. Fracture toughness test specimens with 2.8 x 2.1 x 24 mm³ were cut from the bulk samples as shown in Figure 1, and then annealed in O₂ atmosphere at 723 K for 100 hours. These specimens are denoted as A1-B4, respectively.

A notch was introduced at the center in the longitudinal direction of the specimens as shown in Figure 2 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 0.86-1.23 mm; the reduced notch depth (notch depth/thickness of the specimen) being 0.31-0.44. Load was applied by means of the INSTRON 4464 testing machine (Load cell capacity: 2 kN) at a constant displacement rate of 0.1 mm/min. The fracture toughness $K_{IC}$ was calculated by the following equations (1) and (2) [11].

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

$$Y\left( \frac{a}{W} \right) = 1.964 - 2.837 \frac{a}{W} + 13.711 \left( \frac{a}{W} \right)^2 - 23.250 \left( \frac{a}{W} \right)^3 + 24.129 \left( \frac{a}{W} \right)^4$$ \hspace{1cm} (2)

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. Area fractions and the average sizes of pore and Ag particle on the polished side surfaces with 1.6 x 2.4 mm² were measured for every fractured specimen by using image analysis software.
3. Results and discussion

Figure 3 shows the relationship between the fracture toughness and the Ag content for the Dy123 bulks. The fracture toughness is improved by the Ag addition. The average values are 1.34, 1.44 and 1.54 MPa m$^{1/2}$ for X=5, 7.5 and 10, respectively. This improvement of the fracture toughness by the 5, 7.5 and 10 wt.% Ag additions is similar to the improvement of the bending strength evaluated by using plain specimens [9]. These improvements of the mechanical properties are ascribed to the suppression of crack propagation by ductile Ag particle [4]. Compressive residual stress induced by the difference in the coefficient of thermal expansion between the Dy123 matrix and the Ag particle may be another reason.

Optical micrographs of the top and the inner regions of the Dy123 bulks are shown in Figure 4. The number of pores in the top region is distinctly smaller than that in the inner region. One of the reasons for it is that oxygen bubbles generated near the surface are easily released [12]. On the other hand, there is no significant difference in the distribution of Ag particle between the top and the inner regions for each bulk sample. Although it has been reported that the porosity of Y123 bulks with 0, 10 or 20 wt.% Ag decreases with increase of the Ag content [8], such a behavior is not clearly recognized in association with the small difference in the Ag content among the Dy123 bulk samples in this study as shown in Figure 5. The area fractions of Ag particle for X=7.5 and 10 are close to the volume fractions of Ag particle estimated from the silver compositions. On the other hand, the measured area fraction for X=5 is lower than the estimated volume fraction. This is presumably ascribed to that the size of Ag particle for X=5 is small (see Figure 4) and it is difficult to recognize all the Ag particles in the image processing.

Figure 6 shows the fracture toughness, porosity and area fraction of Ag particle in the specimens. The fracture toughness is excellent near the top surface of the bulks in association with the low porosity. A decrease of the fracture toughness value with increase of the porosity and the average pore size is clearly observed for X=5 as shown in Figures 7 and 8. This is consistent with the fracture toughness behavior of Dy123 bulks without Ag [10], and mainly ascribed to the reduction of the net cross-sectional area of the specimen or to the increase of the net length of the induced V-notch due to the existence of many pores around the notch root. On the other hand, such a dependency is not so clear for X=7.5 and 10, presumably ascribed to the Ag particles with various sizes in these bulk samples (see Figure 4). It has been reported that the cohesive force between the large Ag particle and the bulk material is presumed to be relatively low [13]. Furthermore, it is thought that another reason for it is distributions of pre-existing micro-crack and Dy211 particle in the bulk samples.

The relationship between the fracture strength value and the porosity of ceramics is expressed by the following equation (3) [14].

$$\sigma_f = \sigma_{f0} \exp(-b_1p)$$  \hspace{1cm} (3)

where $\sigma_{f0}$ denotes the fracture strength at 0 % porosity, $p$ is the porosity and $b_1$ is an experimental

![Figure 3. Relationship between fracture toughness and Ag content.](image-url)
constant. Based on the analogy with equation (3), the present authors previously approximated the fracture toughness value at 0 % porosity $K_{IC0}$ of Dy123 bulks without Ag by the following equation (4) [10].

**Figure 4.** Optical micrographs of top (a-1) and inner (a-2) regions for X=5 and top (b-1) and inner (b-2) regions for X=10. Black and white areas indicate pore and Ag particle, respectively.

**Figure 5.** Area fractions of pore and Ag particle in the fracture toughness test specimens.

**Figure 6.** Fracture toughness, porosity and area fraction of Ag particle in the specimens cut from Dy123 bulks with X wt.% Ag.
where $b_2$ is an experimental constant. By performing the extrapolation of data, 1.62 MPa m$^{1/2}$, which is 20 % higher than the average $K_{IC}$ value, and 1.9 were obtained as $K_{IC0}$ and $b_2$ for X=5, respectively. Although it is thought that both of the $K_{IC0}$ value and the average $K_{IC}$ value depend on the bulk sample, it is deduced that the $K_{IC0}$ will be about 20 % higher than the average $K_{IC}$ value for some Dy123 bulks that have a small Ag content and a similar porosity to that for X=5 because the $b_2$ obtained in this study is relatively close to that for Dy123 bulks without Ag obtained in the previous study, 1.6 [10]. However, the estimated $K_{IC0}$ of X=5 is slightly lower than that of Dy123 bulks without Ag, 1.75 MPa m$^{1/2}$, obtained in the previous study [10]. Further investigations associated with the difference in the mechanical properties of the matrix are necessary to clarify the reason for it.

Figure 7 shows the relationship between the fracture toughness value and the area fraction of Ag particle. The fracture toughness value increases with increase of the area fraction of Ag particle. However, such a dependency is not clearly recognized among the specimens for each bulk sample. Since it was considered that the unclear dependency among the specimens for each bulk sample is ascribed to the large scattering of the porosity in comparison with that of the area fraction of Ag particle, the relationship between the estimated $K_{IC0}$ and the area fraction of Ag particle to the area

![Figure 7](image7.png)

**Figure 7.** Relationship between fracture toughness and porosity for Dy123 bulks with X wt.% Ag.

![Figure 8](image8.png)

**Figure 8.** Relationship between fracture toughness and average pore size for Dy123 bulks with X wt.% Ag.

![Figure 9](image9.png)

**Figure 9.** Relationship between fracture toughness and area fraction of Ag particle for Dy123 bulks with X wt.% Ag.

![Figure 10](image10.png)

**Figure 10.** Relationship between estimated fracture toughness at 0 % porosity $K_{IC0}$ and modified area fraction of Ag particle for Dy123 bulks with X wt.% Ag.
excluding pore, which is denoted as modified area fraction of Ag particle \( A_M \), is investigated as shown in Figure 10. The \( K_{IC0} \) was estimated by substituting the evaluated fracture toughness value and the porosity to the equation (4). It is assumed that the \( b_v = 1.9 \) obtained from the extrapolation of data for \( X = 5 \) is applicable to the other bulks with \( X = 7.5 \) and 10. However, the dependency is not still clear. This is also similar to the behavior of the mechanical properties evaluated by using plain specimens [9].

4. Summary
Fracture toughness evaluations on Dy123 single-grain bulks with X wt.% Ag (X = 5, 7.5 and 10) melt-processed in air were carried out at room temperature. The fracture toughness is improved by the Ag addition; the average values are 1.34, 1.44 and 1.54 MPa m\(^{1/2}\) for X = 5, 7.5 and 10, respectively. It is deduced that one of the reasons for it is the suppression of crack propagation by Ag particle. The porosity near the top surface of these bulks is distinctly low and the fracture toughness value is high. In X = 5, an increase of the fracture toughness value with decrease of the porosity is clearly recognized, which is consistent with the behavior of Dy123 bulks without Ag observed in the previous study. On the other hand, such a dependency is not clear for X = 7.5 and 10. According to the extrapolation of data, it is deduced that the fracture toughness value at 0 % porosity will be 20 % higher than the average value.

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