Observations on fracture surfaces of Dy123 bulks with various porosities

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Abstract. Fracture surface observations were carried out for specimens fractured by bending loading. These specimens were cut from Dy123 (DyBa₂Cu₃Oₓ) bulks with various porosities. Fracture surfaces of the oxygen annealed specimens had large steps due to many pre-existing micro-cracks perpendicular to the c-axis. On the other hand, fracture surfaces of the as-grown specimens were macroscopically flat and flow patterns formed by the crack propagations were clearly observed. The fatal crack of some porous specimens originated from pores. On the other hand, the fatal crack of some dense specimens originated from an inclusion. It has been clarified that the inclusion consists mainly of Pt which is added to the precursor to disperse fine Dy211 (Dy₂BaCuO₅) particles in the melt-grown Dy123 bulks.

1. Introduction

Improvement of the mechanical properties of R123 (RBa₂Cu₃Oₓ, where R is yttrium or rare-earth elements) melt-processed bulks is indispensable for the development of superconducting devices equipped with R123 bulk magnets, current leads, current limiters and so on. Melt-processing for R123 bulks is commonly carried out in air or under low O₂ pressure. Conventional R123 bulks melt-processed in air or under low O₂ pressure have pores excepting the region near the surfaces of them [1-3]. Since pores in R123 bulks cause the degradation of the mechanical properties due to the stress concentration and reduction of the net cross-sectional area, elimination of pore is effective in improving the mechanical properties of the bulks. It has been reported that Sm123 bulks melt-processed in 100 % O₂ atmosphere have few pores in the whole region of them [4,5]. Although the mechanical properties of the dense Sm123 bulk were superior to those of a porous Sm123 bulk melt-processed in 1 % O₂ atmosphere [4], the superconducting properties of the former bulk were inferior to those of the latter bulk [5]. Dy123 bulks melt-processed in 100 % O₂ atmosphere also have few pores in the whole region of them [6-9]. Furthermore, the superconducting properties of the dense Dy123 bulk were comparable to those of a porous Dy123 bulk excepting the region just beneath the seed crystal [6]. In the previous studies, we evaluated the mechanical properties of Dy123 bulks with various porosities through bending tests of specimens cut from the bulks [2,3,7-9]. The porosity was decreased by increasing the oxygen pressure in the melt-processing and the mechanical properties were improved [8]. It is thought that fracture surface observations will be informative for further improvement of the mechanical properties of Dy123 bulks. In the present study, observations on the fracture surfaces of specimens cut from Dy123 bulks with various porosities were carried out.
2. Experimental procedure
Bending tests of specimens cut from a dense Dy123 bulk sample which had few pores fabricated by Nippon Steel Corporation were carried out at room temperature. The diameter and the thickness of the bulk sample were 46 and 25 mm, respectively. This bulk sample is denoted as Dy46D as shown in Table 1. The molar ratio Dy123:Dy211 of the precursor of this bulk sample was 75:25. 0.5 wt.% Pt was added to the precursor to disperse fine Dy211 particles in the melt-grown bulk sample [10]. In the previous study, most of the Dy211 particles dispersed in dense Dy123 bulks had the size below 3 μm [9]. Schematic illustration of the melt-processing for the dense Dy123 bulk sample is shown in Figure 1. The precursor was heated in O₂ atmosphere up to 1423 K, kept at that temperature for 1 h and then cooled down to 1313 K. After that, one Nd123 seed crystal was placed on the top of it in air. Bending test specimens with the dimensions of 2.8 x 2.1 x 24 mm³ were cut from the dense bulk sample such that the 2.1 mm direction almost corresponded to the c-axis. The number of the bending test specimens was nine. Oxygen annealing, which is indispensable for the excellent superconducting properties of R123 bulks, was conducted at 723 K for 100 h for three bending test specimens. Three-point bending load in the 2.1 mm direction was applied at 0.1 mm/min by means of INSTRON 4464 testing machine. The loading span was 21 mm. Longitudinal strain caused by the loading was measured through a strain gage glued on the tensile side 2.8 x 24 mm² surface of the specimens. The Young’s modulus was evaluated from the slope of the stress-strain curves. Fracture surfaces were observed by using a scanning electron microscope equipped with an energy dispersive X-ray spectrometer (EDS). Observations on the bending fracture surfaces of specimens cut from other Dy123 bulks were also carried out. Specifications of these bulk samples are also shown in Table 1. The mechanical properties were reported elsewhere [3,8,9]. The porous Dy123 bulks were obtained by heating the precursor in air. The porosity was about 10-20% in the inner region of these porous bulks [3].

Table 1. Specifications of Dy123 bulk samples.

| Sample name | Dy211 [mol%] | Pt [wt.%] | Bulk size [mm] | Dense or porous | Mechanical properties |
|-------------|--------------|-----------|----------------|----------------|----------------------|
| Dy46D       | 25           | 0.5       | φ 46 x 25      | Dense*₁         | Evaluated in this study. |
| Dy30D       | 25           | 0.5       | φ 30 x 10      | Dense*₁         | Reported in Ref. [8]. |
| Dy46P       | 25           | 0.5       | φ 46 x 25      | Porous*₂        | Reported in Ref. [3]. |
| Dy30P       | 25           | 0.5       | φ 30 x 10      | Porous*₂        | Reported in Ref. [3]. |
| Dy3030P     | 30           | 0.5       | φ 30 x 10      | Porous*₂        | Reported in Ref. [9]. |

*1: Dense bulk samples were fabricated by heating precursor in O₂ atmosphere.
*2: Porous bulk samples were fabricated by heating precursor in air.

Figure 1. Schematic illustration of melt-processing for dense Dy123 bulk.
3. Results and discussion

3.1. Mechanical properties of dense Dy123 bulk

The Young’s modulus and the bending strength of the as-grown and the oxygen annealed specimens of the dense Dy123 bulk sample Dy46D are shown in Figure 2. The average values of the other Dy123 bulks [3,8] are also shown for reference. Bending tests of these bulks were carried out under the same conditions. The mechanical properties of the dense bulk Dy46D were superior to those of the porous bulk Dy46P. This improvement of the mechanical properties is attributable to the small stress concentration and large net cross-sectional area in the dense bulk. As mentioned in the followings, the fatal crack of some porous specimens originated from pores where the stress concentration occurs. The Young’s modulus and the bending strength of the dense bulk Dy46D were also decreased by the oxygen annealing as reported for the other dense bulk Dy30D [8]. It is deduced that these decreases are mainly due to the pre-existing micro-cracks induced in the oxygen annealing process. The bending strength of the Dy46D was lower than that of the Dy30D. Since the fatal crack of some dense specimens originated from a Pt inclusion as mentioned in the followings, it is suspected that there is a difference in size or density of the Pt inclusion between the Dy46D and the Dy30D.

(a) Young’s modulus.                                           (b) Bending strength.

Figure 2. Mechanical properties of specimens cut from dense bulk sample Dy46D. The average values of other Dy123 bulks reported in Refs. [3] and [8] are also shown for reference.

3.2. Fracture surface morphology of dense Dy123 bulks

Since fracture surfaces of the oxygen annealed specimens had large steps due to many pre-existing micro-cracks perpendicular to the c-axis formed by the phase transformation from tetragonal to orthorhombic in the oxygen annealing process, flow patterns formed by the crack propagations were not clear. On the other hand, fracture surfaces of the as-grown specimens were macroscopically flat and the flow patterns were clearly observed.

Photographs of a fracture surface of an as-grown specimen of the dense bulk samples Dy46D and Dy30D are shown in Figures 3 and 4, respectively. The bending strengths of these specimens were 113 MPa and 120 MPa [8], respectively. These bending strength values were the maximum value among the as-grown specimens for each bulk sample. The bottom of the fracture surfaces corresponds to the tensile side where the fatal crack was initiated by bending loading. Flow patterns formed by the crack propagations are clearly observed on these fracture surfaces. Fracture surface morphologies of the Dy46D and the Dy30D are similar to each other. The crack initiation site was identified as shown by arrows. Some inclusion was observed on the crack initiation site. It is deduced that the fatal crack originated from the inclusion where the stress concentration occurs due to the mismatch of the stresses induced in the Dy123 bulk and the inclusion. EDS mapping figure around the crack initiation site is shown in Figure 3 (c). Blue parts correspond to the inclusion. It has been clarified that the inclusion consists mainly of Pt which is added to the precursor to disperse fine Dy211 particles in the melt-
grown Dy123 bulks. The size of the Pt inclusion shown by an arrow in Figure 3 (b) is slightly larger than that in Figure 4 (b). This is consistent with the fact that the bending strength of the as-grown specimen of the Dy46D shown in Figure 3 was slightly lower than that of the Dy30D shown in Figure 4. However, it has not been understood at present whether the Pt inclusion size depends on the bulk size or not. In any event, it is expected that suppression of segregation of the Pt inclusion is effective in improving the mechanical properties of dense Dy123 bulks.

Figure 3. (a) Fracture surface of as-grown specimen of dense bulk sample Dy46D, (b) Magnified view around crack initiation site and (c) EDS mapping figure around crack initiation site. Arrows indicate crack initiation site. Bending strength of this specimen was 113 MPa.

Figure 4. (a) Fracture surface of as-grown specimen of dense bulk sample Dy30D and (b) Magnified view around crack initiation site. Arrows indicate crack initiation site. Bending strength of this specimen was 120 MPa [8].
3.3. Fracture surface morphology of porous Dy123 bulks

Large pores of about 300-400μm in size were observed on the fracture surfaces of specimens cut from porous Dy123 bulk samples melt-processed in air. Fracture surfaces of as-grown specimens of the porous bulks also have clear flow patterns formed by the crack propagations. It was clarified that the fatal crack of some as-grown specimens originated from a large pore or the region between two pores. It is deduced that interference of the stress concentration occurred in the region between two pores.

Peculiar fracture surfaces observed for the as-grown specimens of the porous bulk samples are shown in the followings.

Photographs of a fracture surface of an as-grown specimen cut from inner region of the porous bulk sample Dy46P are shown in Figure 5. The bending strength of this specimen was 67 MPa [3], which is equal to the average value of the as-grown specimens of the Dy46P. One pore shown by arrows was identified as the origin of the fracture. This pore is relatively smaller than other pores observed on this fracture surface. However, the Pt inclusion adjoins the pore as shown by an arrow in Figure 5 (b) and (c). It is expected that suppression of segregation of the Pt inclusion is effective in improving the mechanical properties for not only dense Dy123 bulks but also porous Dy123 bulks.

Figure 5. (a) Fracture surface of as-grown specimen cut from inner region of porous bulk sample Dy46P, (b) Magnified view around crack initiation site and (c) EDS mapping figure around crack initiation site. Arrows indicate crack initiation site. Bending strength of this specimen was 67 MPa [3].

Photographs of a fracture surface of an as-grown specimen cut from the region near the top surface of the porous bulk sample Dy3030P are shown in Figure 6. The bending strength of this specimen was 104 MPa [9], which is the maximum value among the as-grown specimens of the Dy3030P. It has been reported that the porosity near the surfaces of porous Dy123 bulks was extraordinarily low [1-3]. One of the reasons for it is that gases in the precursor are easily released [1]. Since bending load was applied such that the tensile side surface of the specimen corresponded to the top surface of the bulk
sample, few pores are observed on the tensile side fracture surface. It was clarified that the fatal crack of this specimen also originated from the Pt inclusion as shown by an arrow in Figure 6 (b).

Figure 6. (a) Fracture surface of as-grown specimen cut from the region near the top surface of porous bulk sample Dy3030P and (b) Magnified view around crack initiation site. Arrows indicate crack initiation site. Bending strength of this specimen was 104 MPa [9].

4. Summary
Fracture surface observations were carried out for specimens cut from Dy123 bulk samples with various porosities. Fracture surfaces of the oxygen annealed specimens had large steps due to many pre-existing micro-cracks perpendicular to the c-axis. On the other hand, fracture surfaces of the as-grown specimens were macroscopically flat and flow patterns formed by the crack propagations were clearly observed. While the fatal crack of some as-grown specimens cut from the porous Dy123 bulks originated from pores, the fatal crack of some as-grown specimens cut from the dense Dy123 bulks originated from an inclusion. It has been clarified that the inclusion consists mainly of Pt which is added to the precursor to disperse fine Dy211 particles in the melt-grown Dy123 bulks. Although it was difficult to observe the flow patterns and identify the crack initiation site on the fracture surfaces of the oxygen annealed specimens, it is expected that suppression of segregation of the Pt inclusion is effective in improving the mechanical properties of Dy123 bulks.

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