Trapped magnetic field of 48 mm diameter single-domain Dy-123 system superconductor

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Abstract. Since melt-textured rare earth (RE)-system superconducting bulk can trap high magnetic field, it acts as a quasi-permanent magnet stronger than conventional permanent magnets. Large-sized single-domain DyBa2Cu3Ox (Dy-123) system bulks about 48 mm in diameter and 15 mm in thickness were prepared with a seeding and temperature gradient method in air. Dy-123 and Dy2BaCuO5 (Dy-211) powders were mixed in molar ratios of 1:0.3. The pellets were partially melted at 1,070°C. When the top temperature of the sample reached 980°C, Nd-123 seed crystal was placed on the top surface of the bulk. Then, the samples were cooled from 980°C to 930°C, and then with furnace cooling. Finally, the samples were annealed in pure oxygen atmosphere. When magnetic field of 1.5 T was applied to the samples, the maximum trapped magnetic field of 0.7 T was trapped at 77.3 K in the samples prepared with the cooling time for 600 hr and 700 hr from 980°C to 930°C. Only 0.2 T was trapped in the sample prepared with the cooling time for 1,000 hr, whose bottom part was decomposed due to long time heating.

1. Introduction
The melt-textured DyBa2Cu3Ox (Dy-123) system superconductors which were grown by means of a top seeding and temperature gradient method have high potential for superconductivity applications, since they exhibit higher critical current density ($J_c$) in high magnetic field compared to melt-textured YBCO superconductors [1-4]. It was reported that high magnetic field of 1.9 T was trapped at 77.3 K in the presence of 7 T applied parallel to the c-axis for Dy-123 bulk superconductor [5]. A large-sized single-domain Dy-123 Superconductor, therefore, is expected for large-scale applications, flywheels, magnetic bearings, magnetic separation, etc.

In the previous papers [6-8], superconducting properties of melt-textured bulk of Dy-123 system with different molar Dy-123/Dy-211 ratios was studied. Here, both $J_c$ and trapped magnetic field increased with the amount of Dy-211. And single-domain bulks, 48 mm in diameter and 15 mm in thickness, were prepared in air. When magnetic field of 1.5 T was applied to the sample, 0.7 T was trapped at 77.3 K. In this report, we investigated the effect of the cooling rate from 980°C to 930°C on
the superconductivity of the single-domain samples 48 mm in diameter fabricated under various conditions.

2. Experimental

2.1. Sample preparation

Dy-123 and Dy-211 powders were mixed in the molar ratio of 1/0.3[7]. Since Ag$_2$O addition is effective in lowering the melting point and improving the mechanical strength, Ag$_2$O powder of 10 wt% was added to the mixture. Then, Pt powder of 0.5 wt% was added to the mixture. The mixed compounds were molded with metal dies and pressed into pellets using coaxial pressing equipment. The prepared pellet was 52 mm in diameter and 15 mm in thickness. In addition, we prepared the same component pellet without Ag$_2$O, 52 mm in diameter and 5 mm in thickness. This pellet was placed under the sample pellet during melt-solidification process instead of Al$_2$O$_3$ plate. The reason is that, when Al$_2$O$_3$ plate is used as a base plate, aluminum atoms diffuse into the sample, which deteriorates the superconducting property of the sample.

The sample pellets were sintered at 900°C for 24 hours in air. The superconducting bulks were fabricated with the top seeding method under temperature gradient in air. The pellets were partially melted at 1,070°C for 4 hour, and rapidly cooled to 980°C in one hour. Here temperature gradient of 10°C/cm was applied by heating from the bottom to the surface. When the top temperature of the sample reached 980°C, the $ab$-plane of Nd-123 seed crystal was placed in contact with the top surface of the bulk to grow a grain along the $c$-axis. After the seeding, the samples were cooled with a different cooling rate with 0.083°C/hr, 0.072°C/hr and 0.05°C/hr, and three bulks (A, B and C) processed with the cooling time of 600 hr, 700 hr and 1,000 hr from 980°C to 930°C were obtained, respectively. Then, the bulks were cooled to room temperature with furnace cooling. Finally, the samples were annealed in pure oxygen atmosphere from 500°C to 250°C for 500 hr. In all the cases, the size of the bulks was obtained 48 mm in diameter.

Figure 2. Photographs of single-domain Dy-123 melt-textured bulks 48 mm in diameter. (a) bulk A, (b) bulk B and (c) bulk C.
2.2. Measurement

The structure characterization in single-domain Dy-123 system superconductor was performed using X-ray diffraction (XRD) and scanning electron microscopy (SEM). For evaluation of superconducting properties of the bulk, we measured magnetization curves of the specimens cut from the bulks. Critical temperature \(T_c\) was estimated from temperature dependence of the magnetization and \(J_c\) in self-field was calculated using the Bean model. The trapped magnetic field \(B_T\) at 77.3 K of the superconductors was measured after applying magnetic field of 1.5 T by placing a hall sensor onto the sample surface. Figure 1 shows a schematic diagram of the specimens cut from a massive bulk sample for measurement of XRD, SEM, \(T_c\) and \(J_c\).

3. Results and Discussion

3.1. Superconducting Property

Figures 2 (a), (b) and (c) show photographs of single-domain of Dy-123 melt textured bulks (A, B and C) 48 mm in diameter. It is observed that the domain grows from Nd-123 seed crystal to the edge in the surface of the bulk.

Figures 3 (a), (b) and (c) show the trapped magnetic field distributions at 77.3 K for bulks (A, B and C), respectively. Almost single peaks of the trapped magnetic field mean that fabrication of Dy-123 system melt-textured bulks by means of the top seeding and temperature gradient method was succeeded. The maximum trapped field is 0.6-0.7 T at the center of the bulk A and bulk B with the cooling time for 600 hr and 700 hr from 980°C to 930°C. In the bulk C prepared with the cooling time for 1,000 hr, 0.2 T of the maximum trapped field is observed.

Figure 4 shows the change of \(J_c\) value at 77.3 K in the bulks. The \(J_c\) decreases from the upper part to the bottom part of the bulks, and the \(J_c\) values in the edge region are higher than those in the center.
region of the bulks. In Figure 4 (c), the $J_c$ of the bottom part in the center region of the bulk C is low value, $1 \times 10^3$ A/cm$^2$.

Figure 4. $J_c$ of specimens at 77.3 K cut from Dy-123 melt texture bulk. (a) bulk A, (b) bulk B, (c) bulk C. □; center region and △; edge region. Close symbols are mean value.

Figure 5 shows the change of $T_c$(onset) and $T_c$(offset) value from the temperature dependence the magnetization of the specimens cut from the center region of the bulks (A, B and C). The $T_c$ decreases from the upper part to the bottom part of the bulks, and the $T_c$(offset) of the bottom part of the bulk C is 90.8 K.

It is mentioned that the $J_c$ and $T_c$ property of the Dy-123 melt texture bulk deteriorates in the bottom parts, which is influenced by the cooling rate from 980°C to 930°C during domain growth.

3.2. Microstructure

Results of back-scattering electron imaging of SEM taken on the surface of specimens cut from Dy-123 melt textured bulks (A and C) are shown in Figures 6 (a) and (b), respectively. In these images, the grey matrix region is Dy-123 phase and small white particles are Dy-211 phase. The Dy-211 particles are dispersed homogeneously in the Dy-123 phase matrix. In Figure 6 (a), large white regions of a few ten μm is metallic Ag are observed in the sample prepared with cooling rate of 600 hr. Figure 6 (b) shows that there are scarcely observed Ag regions in the sample prepared with cooling time for 1,000 hr, and in the bottom parts ((3), (6)) dark gray region and needle like structure are observed. As result of average of energy dispersive x-ray analysis (EDX), CuO or Cu rich compounds was assigned.
in the dark gray region, and the needle like structure was comprised of Dy, Ba, Cu elements but the composition has not yet determined.

Figure 6. SEM photographs of specimens cut from Dy-123 melt textured bulk. (a) bulk A and (b) bulk C. Numbers in the photographs correspond with specimens in Figure 1.

Figure 7 shows XRD patterns of specimens cut from the center region of bulk C. Strong (00\(l\)) peaks at about 23\(^\circ\), 38\(^\circ\) and 46\(^\circ\) of 2\(\theta\) indicate that the c-axis oriented Dy-123 phase grow from the upper surface to the middle part reflecting the grain axis of the Nd-123 seed crystal. Dy-211 peaks at around 30\(^\circ\) are observed in all specimens. In the bottom part of the bulk, instead of almost disappearance of the Dy-123 phase, a peak at around 33\(^\circ\) appears, which has not yet assigned but
related probably to CuO or Cu compounds.

It is suggested from results of SEM photographs and XRD diffraction patterns that in the bulk C prepared with cooling rate of 1,000 hr, Dy-123 phase decomposes to CuO or Cu compounds in the bottom part.

4. Conclusions

Three samples of single-domain Dy-123 system superconductor bulks, 48 mm in diameter and 15 mm in thickness were prepared with the seeding and temperature gradient method in air. When a magnetic field of 1.5 T at 77.3 K was applied to the bulks prepared with the cooling time for 600 hr and 700 hr from 980°C to 930°C, the maximum trapped field of 0.6 – 0.7 T was trapped. The $J_c$ decreased from the upper part to the bottom part of the bulk. It was mentioned that the bottom part was deteriorated with Dy-123 phase decomposition from the result of the SEM photograph and the XRD measurement.

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