Evaluation of Microstructure, Resistance, and Critical Current of REBCO Superconducting Joints Fabricated by Slurry Process

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***Abstract*—The properties of superconducting joints for $REBa_2Cu_3O_y$ (REBCO, RE = rare earth) superconducting tapes fabricated using a slurry process were investigated. Microstructural observation revealed that a joining layer fabricated using a slurry was well-connected to the REBCO layer of the joined tape. The resistance and critical current of closed-loop samples containing slurry-processed superconducting joint were evaluated using current decay measurements. The joint resistance was found to be lower than 10^{-12} and 10^{-11} Ω at 4 and 77 K, respectively, in the self-field. It is inferred that a persistent current can flow through a slurry-processed superconducting joint. Conversely, in-field critical current was low and exhibited hysteresis due to weak links in the joining layer.**

***Index Terms*—2G HTS conductors, coated conductors, resistance measurement, critical current**

I. Introduction

The development of superconducting joints between REBCO coated conductor tapes for operation of REBCO superconducting magnets in the persistent mode has seen significant progress [1][2][3]. To fabricate superconducting joints, the exposed REBCO thin film layers of tapes are joined via a newly formed REBCO layer through REBCO crystal growth at the joining interface [4][5][6][7][8]. Epitaxial growth of the REBCO thin film layer is the most common process used for joining. This can help obtain superconducting joint samples with resistances below 10^{-12} Ω at currents of 10^1–10^2 A.

The joining methods developed so far require further improvement [3]. In particular, the epitaxial growth method poses technical challenges to the joining process. This is because to join the REBCO layers at the atomic level, the *ab*-plane and the *c*-axis of the two layers must be aligned within a few degrees of misorientation. Considering that joining will routinely be performed at manufacturing facilities of superconducting magnets, an easy-to-use joining method must be established.

Recently, we have been developing a method for joining REBCO tapes using a slurry [9]. A polycrystalline joining layer is fabricated using a slurry between the exposed REBCO layers. This method is technically simple because it does not require epitaxial growth. In addition, slurry-processed joints with compact sizes and a variety of shapes that are suitable for implementation in persistent-mode magnets can be fabricated owing to few restrictions on the shapes of the joining layer and joint configurations.

In this study, we evaluated the microstructure, joint resistance (R_j), and joint critical current (I_{cj}) of slurry-processed REBCO superconducting joints. Loop samples closed with the slurry-processed joint were fabricated using a REBCO tape. R_j and I_{cj} of the joints were evaluated by performing current decay measurements. Following the measurements, the microstructure of one of the joints was observed.

II. Experimental

A. Preparation of closed-loop samples

Fig. 1(a) shows a schematic of the fabricated joint structure. To demonstrate a slurry-processed joint, we prepared a bridge with a joining layer. The use of a bridge was effective in decomposing organic components in a slurry [6]. That is, we coated a slurry onto a bridge and then calcined the coated bridge at high temperatures for the decomposition. The closed-loop sample consisted of a 1 m long and 4 mm wide REBCO tape and a 12 mm wide REBCO tape for the bridge. EuBCO tapes with $BaHfO_3$ artificial pinning centers (Fujikura FESC-S04 and FESC-S12) [10] were used as the REBCO tapes. Both tapes did not have a copper-stabilizing layer. According to the inspection report, the critical current of the 4 mm wide tape at 77 K in the self-field was 204 A with a 10^{-6} V cm^{-1} criterion.

To expose the REBCO layer, the silver layer was removed from both ends of a 4 mm wide tape and the entire bridge by chemical etching. The exposed REBCO layers were to be placed facing each other. To form a joining layer between the opposing REBCO layers, a slurry was deposited by dip-coating onto the REBCO layer of the bridge. The slurry was prepared by mixing EuBCO powder (TEP Co, Ltd.) and a metal-organic deposition (MOD) solution (Kojundo Chemical

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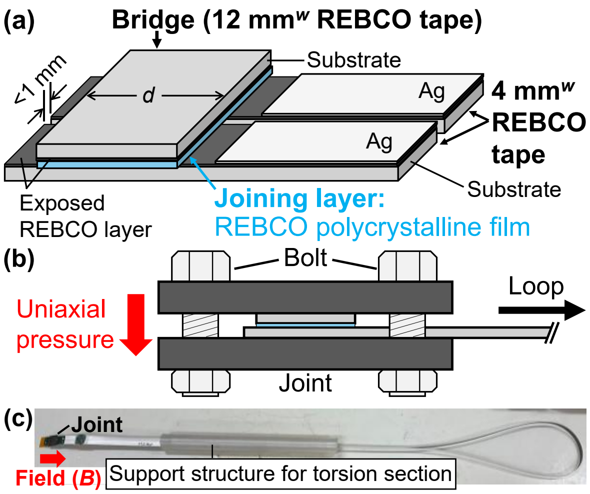

Fig. 1. (a) Schematic of the structure of the slurry-processed joint. (b) Schematic of the side view during the heat treatment with applying a uniaxial pressure to the joint. The pressure was controlled by bolt-tightening torque. (c) Photograph of a single-turn REBCO closed-loop sample with the joint. Magnetic field (B) is applied to the joint as shown in the figure.

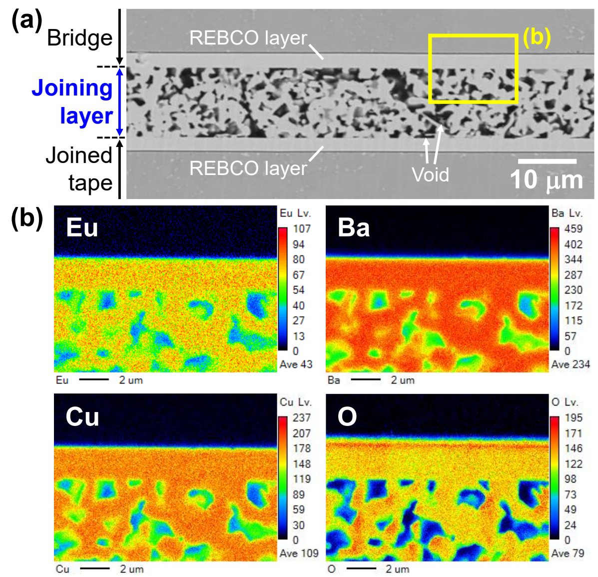

Fig. 2. (a) Backscattered electron image of polished surface of cross-section of joint in #1. (b) EPMA elemental maps for Eu, Ba, Cu, and O. The evaluated region is shown in (a).

Laboratory Co., Ltd.). To decompose organic components and form a microcrystalline layer on REBCO grains from the MOD solution [3][6], the coated bridge was calcined at 840°C in oxygen flow. We made the joint configuration using the calcined bridge. The joining part was fixed by bolt-tightening, as shown in Fig. 1(b). This setup enables the application of uniaxial pressure to the joint during the heat treatment to form a superconductive joining layer. After a heat treatment at 840°C in 0.05%O_2/Ar flow and annealing at 450°C in oxygen flow, the bolts were loosened to release the applied pressure for the characterizations.

We fabricated four single-turn closed-loop samples, labeled as #1, #2, #3, and #4. Fig. 1(c) shows a photograph of #1. For the current decay measurements, we fixed the torsion section using a support structure [11]. The loop mounted on the sample holder had a diameter of 100 mm. The self-inductance (L) of the sample was 4.7×10^{-7} H, which was evaluated using a loop sample with the same shape in our previous study [12].

Table I lists the fabrication conditions for the slurry-processed joint of the samples. The bridge length (d) is defined as shown in Fig. 1(a). In a preliminary experiment, using a pressure measurement film (Fujifilm Prescale), we estimated the uniaxial pressure applied to the joint by the bolt-tightening torque of 2.0 N m to be about 3–5 × 10^7 Pa (30–50 MPa) at room temperature. Considering that the coefficient of thermal expansion of stainless-steel bolts [13] is higher than that of the Hastelloy C-276 substrate [14], the applied pressure at 840°C would be lower than that at room temperature.

B. Evaluation of closed-loop samples

Current decay measurements were performed for the samples at 4 and 77 K using a joint resistance evaluation system [12]. A magnetic field (B) of 0.02–1 T was applied to the joint for in-field measurements. The direction of the field is illustrated in Fig. 1(c). This direction was fixed in the evaluation system used. The decay of the loop current (I_{loop}), that is, the time (t) dependence of I_{loop} was measured at a sampling rate of 1 Hz. We introduced I_{loop} into the sample through magnetic induction using a copper coil (injection coil) located at the center of the loop. The initially introduced I_{loop} was controlled by an injection coil current (ICC). The initial I_{loop} value was about 20 times ICC.

To evaluate R_j, the experimentally obtained I_{loop}–t data points were fitted to the equation of $I_{loop}(t) = I_{loop}(0)\exp\left(-\left(R_j/L\right)t\right)$ [15][16]. To determine I_{cj}, the voltage of the joint (V) was calculated from the I_{loop}–t data points using $V = -L\left(\Delta I_{loop}/\Delta t\right)$. From the obtained V–I_{loop}, I_{cj} was determined under a voltage criterion (V_c) of 10^{-8} V [17].

After the current decay measurements, the microstructure of the joint in sample #1 was evaluated. The polished surface of the cross-section of the joint was observed using an electron probe microanalyzer (EPMA, JEOL JXA-8530F Plus). We obtained a backscattered electron image and corresponding elemental maps, as shown in Fig. 2.

TABLE I
FABRICATION CONDITIONS OF THE JOINT FOR THE CLOSED-LOOP SAMPLES

Sample	Bridge length, d (mm)	Bolt-tightening torque (N m)
#1, #2	10	2.0[1)]
#3, #4	15	6.0

[1)]The pressure applied to the joint at room temperature was estimated to be about 3–5 × 10^7 Pa (30–50 MPa) in a preliminary experiment.

III. RESULTS AND DISCUSSION

A. Microstructure of the slurry-processed joint in #1

Fig. 2(a) shows a backscattered electron image of the polished surface of the joint cross-section in sample #1. The upper and lower parts correspond to the bridge and the joined

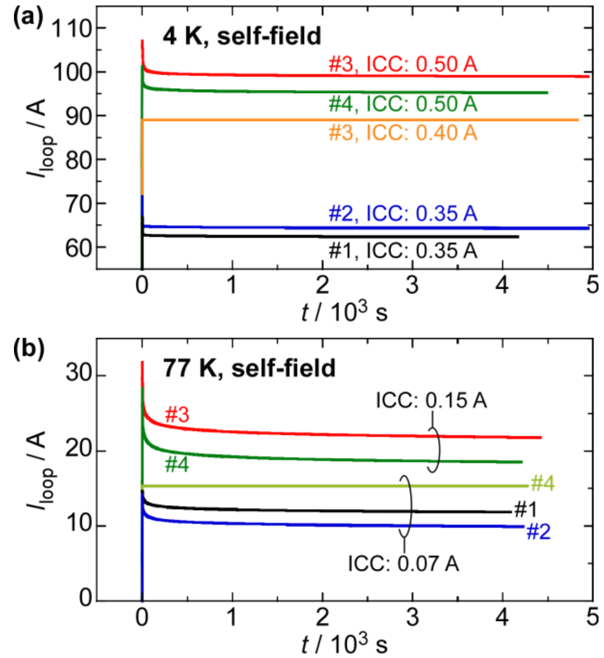

Fig. 3. Time dependence of I_{loop} for the samples #1–4 in the self-field at (a) 4 K and (b) 77 K. Initially introduced I_{loop} was controlled by injection coil current (ICC). A slow decay of I_{loop} was observed in all samples at 4 and 77 K. This implies that a persistent current can flow in the slurry-processed joint. Samples #3 and #4 showed higher I_{loop}, corresponding to higher I_{cj}.

tape, respectively. The central region corresponds to the joining layer. It is observed that the REBCO layers of the bridge and tape are dense and do not exhibit any cracks or decomposition. This implies that degradation of the REBCO layer due to the thermomechanical load, as previously reported in [18], is negligible. In the joining layer, many voids corresponding to the black region are observed, implying that the joining layer has a porous microstructure. However, REBCO grains appeared to be well-connected to each other and to both REBCO layers. This is probably attributed to the slight grain growth by the microcrystalline layer on the grains during the heat treatment.

Fig. 2(b) shows the EPMA elemental maps of Eu, Ba, Cu, and O at the joining interface between the bridge and the joining layer shown in Fig. 2(a). The chemical composition of the joining layer is comparable to that of the REBCO layer of the bridge. We observed no cracks or secondary phases at the joining interface. At this magnification, the grains of the joining layer appeared to be well-connected to the REBCO layer. As the grains are most likely misoriented with respect to the REBCO layer, further microstructural evaluation is required to clarify the connection state.

B. *Evaluation of self-field characteristics of the joints*

Figs. 3(a) and 3(b) show the time dependence of I_{loop} for samples #1–4 in the self-field at 4 and 77 K, respectively. Table II summarizes the self-field I_{cj} and R_j at 4 and 77 K. In most results shown in Fig. 3, $I_{loop} > I_{cj}$ was introduced at $t = 0$, resulting in a fast current decay observed at $t < 10^2$ s. From this fast decay, I_{cj} was determined. After the fast decay, a slow decay of I_{loop} was observed. In these measurements, R_j was evaluated using the data points at 3.5–4.0 × 10^3 s. The low R_j values of 1.7–2.9 × 10^{-13} Ω at 4 K and 2.3–3.4 × 10^{-12} Ω at 77 K correspond to persistent currents flowing in the samples. This implies that a persistent current can flow through the slurry-processed joint in the superconducting state. The higher R_j values at 77 K can be attributed to thermally activated flux motion at the joint, as implied in our previous study [19].

As demonstrated for #3 at 89 A in 4 K and for #4 at 15 A in 77 K, when $I_{loop} < I_{cj}$ was introduced, flatter I_{loop}–t curves were observed. In these measurements, R_j was evaluated using the data points at 2.0–4.0 × 10^3 s based on the lower signal-to-noise ratio. Similar to previous studies, lower R_j values were observed owing to the lower load factor [11][17][19][20].

Samples #3 and #4 showed higher I_{cj} values than #1 and #2, indicating that modification of the fabrication conditions was effective for increasing I_{cj}. The longer bridge length for #3 and #4 increased the effective joining area. The higher bolt-tightening torque for these samples likely produced higher pressure to the joint during the heat treatment, resulting in the formation of a denser joining layer.

TABLE II
CRITICAL CURRENT AND RESISTANCE OF THE JOINT FOR THE SAMPLES IN THE SELF-FIELD AT 4 AND 77 K

Sample	4 K, self-field		77 K, self-field	
	I_{cj} (A)	R_j (Ω)	I_{cj} (A)	R_j (Ω)
#1	63	1.7 × 10^{-13} (62 A)$^{2)}$	14	2.3 × 10^{-12} (12 A)$^{2)}$
#2	65	1.8 × 10^{-13} (64 A)$^{2)}$	12	3.4 × 10^{-12} (10 A)$^{2)}$
#3	101	2.9 × 10^{-13} (99 A)$^{2)}$ 1.2 × 10^{-14} (89 A)$^{3)}$	24	3.0 × 10^{-12} (22 A)$^{2)}$
#4	97	2.7 × 10^{-13} (95 A)$^{2)}$	22	3.1 × 10^{-12} (19 A)$^{2)}$ 7.9 × 10^{-15} (15 A)$^{3)}$

$^{2)}$ R_j was evaluated using the data points at 3.5–4.0 × 10^3 s.
$^{3)}$ R_j was evaluated using the data points at 2.0–4.0 × 10^3 s.

C. *Evaluation of in-field characteristics of the joints at 4 K*

We evaluated the in-field I_{cj} values at 4 K in the magnetic-field range of 0–1 T. Fig. 4 shows the magnetic field dependence of I_{cj} at 4 K for #1, #3, and #4. Rapid decreases in I_{cj} were observed in these samples with increasing fields up to 0.1 T. This implies that the presence of weak links in the polycrystalline joining layer, similar to a polycrystalline bulk [21][22]. By contrast, as the field increased from 0.1 to 1 T, the decreases in I_{cj} in #3 and #4 were relatively small.

To investigate the presence of weak links in the joining layer, we attempted to observe the hysteresis of the in-field I_{cj}. Fig. 5 shows the magnetic field dependence of I_{cj} for #3 at 4 K. We evaluated I_{cj} as the field was increased from 0 to 0.3 T, as shown by the open symbols. We also evaluated I_{cj} as the field was decreased from 0.1, 0.2, and 0.3 T to 0, as shown by the closed symbols. For all measurements, the in-field I_{cj} exhibited hysteresis, demonstrating that the width of the hysteresis increased with the maximum applied field. This suggests the

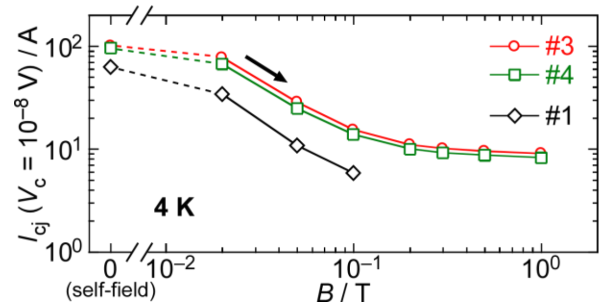

Fig. 4. Magnetic field dependence of I_{cj} for the samples #1, #3, and #4 at 4 K with increasing the field. The I_{cj} values at B = 0 correspond to self-field I_{cj} shown in Table II.

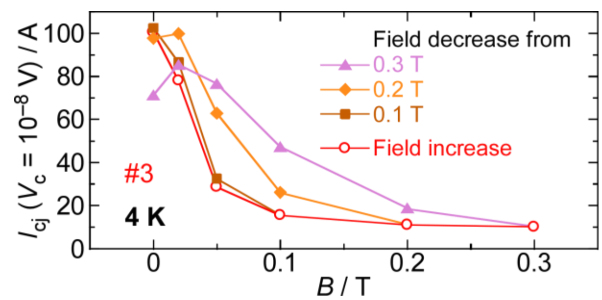

Fig. 5. Magnetic field dependence of I_{cj} for #3 at 4 K with increasing and decreasing the field. In-field I_{cj} exhibited hysteresis. This suggests the presence of weak links of the joining layer.

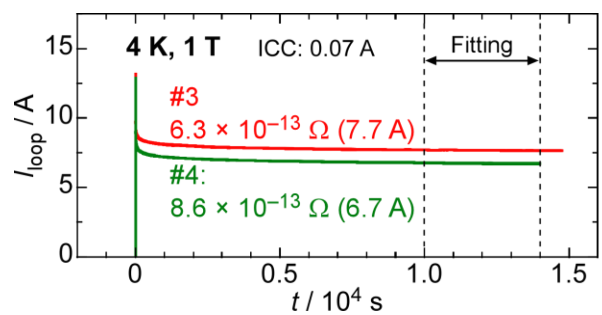

Fig. 6. Time dependence of I_{loop} for #3 and #4 at 4 K and 1 T. Flat curves were observed at t > 10^4 s, corresponding to the persistent current in the samples.

presence of weak links due to the polycrystalline nature of the joining layer [22][23].

Fig. 6 shows the time dependence of I_{loop} for samples #3 and #4 at 4 K and 1 T. By applying a magnetic field of 1 T, I_{loop} decreased significantly compared to that in the self-field, as shown in Fig. 3(a). This corresponds to a decrease in I_{cj} upon field application, as shown in Fig. 4. However, flat curves were observed at $t > 10^4$ s. As shown in Fig. 6, the R_j values at 4 K and 1 T for #3 and #4 obtained using the data points at 1.0–1.4 × 10^4 s were 6.3 × 10^{-13} Ω at 7.7 A and 8.6 × 10^{-13} Ω at 6.7 A, respectively. These R_j values are comparable to those in the self-field, as listed in Table II. These results imply that even in magnetic fields, a persistent current can flow through the slurry-processed joint in the superconducting state.

D. Discussion and future work

As shown in Fig. 2, the REBCO layer of the tape is well-connected to the grains in the joining layer at the interface. This microstructure allows a persistent current to flow through the slurry-processed joint in the superconducting state. However, I_{cj} must be improved, particularly in magnetic fields. The I_{cj} value was notably lower than the critical current of the original tape and that of the superconducting joints fabricated using epitaxial growth [4][6][7][8]. We attempted to estimate rough critical current ratio (CCR: I_{cj} divided by tape I_c) [2] at 77 K in the self-field. We used the I_{cj} (V_c = 10^{-8} V) and tape I_c at 10^{-8} V cm^{-1} (162 A), which is extrapolated from that at 10^{-6} V cm^{-1} using a power law and a typical n value of 20 [1]. The rough CCR value was estimated to be only 7.4–15%.

As implied in this study, an increase in the effective joining area can be a promising approach for increasing I_{cj}. Additionally, the formation of a denser joining layer can also be effective in obtaining higher I_{cj}. This densification effect is consistent with that observed in our previous study of $(Bi,Pb)_2Sr_2Cu_2Cu_3O_y$ (Bi-2223) superconducting joints [24]. In the following study, we plan to investigate the effect of the joining area and densification on I_{cj} in the slurry-processed joint. By contrast, another strategy may be required to increase the in-field I_{cj}. Considering the random orientation of grains of a joining layer, grain alignment may be one of the approaches to increase the in-field I_{cj} [22].

We found that the samples fabricated under the same conditions exhibited similar I_{cj} and R_j characteristics. This implies the relatively high reproducibility of the fabrication of the slurry-processed joints. Such high reproducibility is in part due to the lack of requirement for precise alignment of the c-axis of the REBCO layers and the direction of the applied uniaxial pressure. Therefore, the slurry process may be suitable for an on-site joining method.

Various joint configurations can be achieved using the slurry process. In principle, a joining layer can be directly formed between REBCO layers without a bridge. This will allow fabrication of joints with compact sizes or shapes suitable for implementation in persistent-mode magnets.

It is necessary to investigate the field angular dependence of I_{cj} and R_j, that is, the dependence of I_{cj} and R_j on the magnetic field direction. In previous studies, the angular dependence of I_{cj} and R_j for Bi-2223 and REBCO superconducting joints was evaluated [17][25][26]. Both I_{cj} and R_j exhibited strong angular dependence, reflecting an anisotropic microstructure. By contrast, the slurry-processed joint will exhibit flat angular dependence of I_{cj} and R_j owing to the random orientation of the grains of the joining layer. Such flat angular dependence will be a considerable advantage of slurry-processed superconducting joints.

Considering electromagnetic forces applying to the joints in persistent-mode magnets, the mechanical properties of the slurry-processed joints should be evaluated. If the mechanical strength is insufficient due to the brittleness of the porous joining layer, reinforcement may need to be considered [3].

IV. Conclusion

We developed and evaluated slurry-processed REBCO superconducting joints. Microstructural observations showed that the polycrystalline joining layer fabricated using the slurry was well-connected to the REBCO layer of the tape. The joint resistance was evaluated to be low based on current decay measurements. The critical current rapidly decreased with increasing magnetic field. The in-field critical current exhibited hysteresis due to weak links in the joining layer. Further studies are needed to demonstrate the considerable advantages of slurry-processed joints for implementation in persistent-mode magnets.

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