

Fabrication of Bi-2223 Superconducting Joint by Hot-Pressing Process

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Abstract—Superconducting joints between Bi-2223 tapes were fabricated by a hot-pressing process with a 24 h heat treatment. We developed specialized equipment to apply pressure to the joint during the heat treatment. Hot pressing is effective for producing practical critical current even by the short heat treatment. The superconducting joint in a closed-loop showed a resistance of $7.3 \times 10^{-15} \Omega$ at 4.2 K in self-field. The maximum persistent current that could flow in the loop was estimated to be about 170 A at 4.2 K. The purification and further densification of the intermediate layer in the superconducting joint are issues that need to be addressed. Once these issues are addressed, the hot-pressing process should be a promising method for fabricating high-performance Bi-2223 superconducting joints in high yields within a short processing time.

Index Terms— Bi-2223 tape, HTS magnets, resistance measurement, critical current

I. INTRODUCTION

PERSISTENT current mode superconducting magnets are used for magnetic resonance imaging (MRI) and nuclear magnetic resonance (NMR). Superconducting joints are indispensable for persistent-mode magnets [1], [2]. To realize persistent-mode magnets using high-temperature superconducting (HTS) tapes/wires, the formation of superconducting joints between the HTS tapes/wires is necessary, and significant progress has been made in this area in the last decade [2]–[11].

Ag sheathed multifilamentary $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$ (Bi-2223) HTS tapes are commercially available. The most famous is DI-BSCCO[®] produced by Sumitomo Electric Industries, Ltd. [12], [13]. This tape shows a high critical current (I_c) and has been used for various magnets generating high fields or operating at high temperatures [13]–[16]. However, practical persistent-mode Bi-2223 magnets have not yet been realized, owing to the lack of superconducting joint formation technology.

Manuscript received xxxxxxxx; accepted xxxxxxxx. Date of publication xxxxxxxx; date of current version xxxxxxxx. This work was supported by JST-Mirai Program Grant Number JPMJMI17A2 and JSPS KAKENHI Grant Number JP22K14482, Japan (*Corresponding author: Yasuaki Takeda.*)

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We have previously developed superconducting joints with a high I_c between the Bi-2223 tapes [3], [4]. In the formation of the superconducting joints, most of the Bi-2223 filaments were exposed by low-angle (0.3°) polishing using a tool with a 0.3° tilted surface that we made. To connect the exposed filaments, we synthesized an intermediate layer, which was equivalent to an about 0.1 mm thick polycrystalline Bi-2223 film. This thick film was synthesized through the slurry process, uniaxial pressing at room temperature (RT), and heat treatments. So far, we achieved an I_c of about 300 A at 4.2 K and 1 T using a short sample of the Bi-2223 superconducting joint [4]. We also fabricated a Bi-2223 closed-loop sample with the superconducting joint under the same condition as that of the short sample. Using this closed-loop sample, a low joint resistance (R_j) of less than $10^{-14} \Omega$ at 4.2 K and 1 T was demonstrated [4].

In a recent study [17], we reported that the densification of the intermediate layer is effective in increasing the I_c of Bi-2223 superconducting joints. This increase in I_c was attributed to the increase in the intergrain critical current density (J_c) of the intermediate layer. The densification was achieved by introducing the general two-step heat treatment process. This process comprises the first heat treatment, an intermediate uniaxial pressing at RT, and the second heat treatment. Because of this process, the filling factor of the intermediate layer could be increased to about 80%. However, high-pressure intermediate pressing at pressures more than 2×10^8 Pa (about 200 MPa) can mechanically damage the Bi-2223 filaments in the tapes, which decreased the I_c of the superconducting joints. This suggests that methods other than the intermediate pressing are required to further densify the intermediate layer and improve I_c . A promising densification method is hot pressing.

It is well known that polycrystalline Bi-2223 materials swell with the grain growth during a heat treatment, resulting in a decrease in the density [18], [19]. Hot pressing is effective in suppressing this swelling [20], [21]. It has been reported that the I_c of Bi-2223 tapes increased by introducing a hot-pressing process at pressures of $1\text{--}6 \times 10^6$ Pa.

Attempts to fabricate Bi-2223 superconducting joints by hot pressing have already been reported [22], [23]. The superconducting joints appeared to be formed by hot pressing even with a short heat treatment of about 10 h, although the high-purity Bi-2223 intermediate layer was not synthesized. An R_j value of less than $10^{-13} \Omega$ at 77 K was observed in a Bi-2223 closed-loop sample made from a DI-BSCCO[®] tape. However,

practical persistent current values of more than 10^2 A have not yet been realized.

In this study, we fabricated high-performance Bi-2223 superconducting joints using a hot-pressing process. A short heat treatment was performed to reduce the processing time, making the proposed process a practical one for forming joints.

II. EXPERIMENTAL

A. Preparation of hot-pressing equipment (HOPE)

Specialized equipment for hot pressing, which we have labelled HOPE (HOt-Pressing Equipment). The equipment had to be smaller than the inner diameter of a tube furnace (40 mm). Fig. 1(a) shows a photograph of a straight lap joint sample with HOPE before it was subjected to a heat treatment. HOPE consists of two stainless-steel (SS) plates ($3.0 \times 20 \times 25$ mm) with four holes, four M4 brass bolts, and four alumina nuts. Uniaxial pressure perpendicular to the tape surface was applied on the joint by the axial force of the bolts. The pressure was controlled by the bolt-tightening torque (N) of 0.20 or 0.50 N·m.

The surfaces of the SS plates were oxidized in advance by annealing in air at 800°C to control the partial oxygen pressure (P_{O_2}) during the heat treatment. The SS plates and alumina nuts were durable and could be repeatedly used. However, the brass bolts had to be disposed owing to the oxidization of their surfaces during the heat treatment. We used the brass bolts because the linear thermal expansion coefficient of brass is similar to that of silver, which is the matrix of the Bi-2223 tapes [24]. We believe that by using the brass bolts, the pressure applied at RT could be maintained during the heat treatment at a holding temperature of 820°C .

B. Fabrication of samples by hot-pressing process

Four samples (three straight lap joint samples and one loop sample) were fabricated using the Bi-2223 tapes without mechanical reinforcements (DI-BSCCO® type H, 4.2 mm wide and 0.22 mm thick). To apply a uniform uniaxial pressure, we flattened the tapes by polishing both of their sides [17].

For each straight lap joint sample about 10 cm long (SJ-0, SJ-02, and SJ-05), two 6 cm long tapes were used, as in [3] and [17]. The loop sample (CL-02) was a three-turn closed-loop with a praying-hands type superconducting joint using one 1.6 m long tape, similar to the sample in [4]. The diameter of the loop was 100 mm. The self-inductance (L) of CL-02 was estimated to be $1.4 \mu\text{H}$.

The joining processes of exposing the filaments, the slurry process, and uniaxial pressing at RT were the same as that used in our previous study. The overlap length was 16–17 mm shown in Fig. 1(b). The area of the Bi-2223 intermediate layer was about 70 mm^2 . Before the heat treatment, uniaxial pressing on the joint was performed at 2×10^8 Pa and RT to densify the intermediate layer.

Schematic of the fabrication procedure for the closed-loop sample CL-02 is shown in Fig. 1(c). During the heat treatment

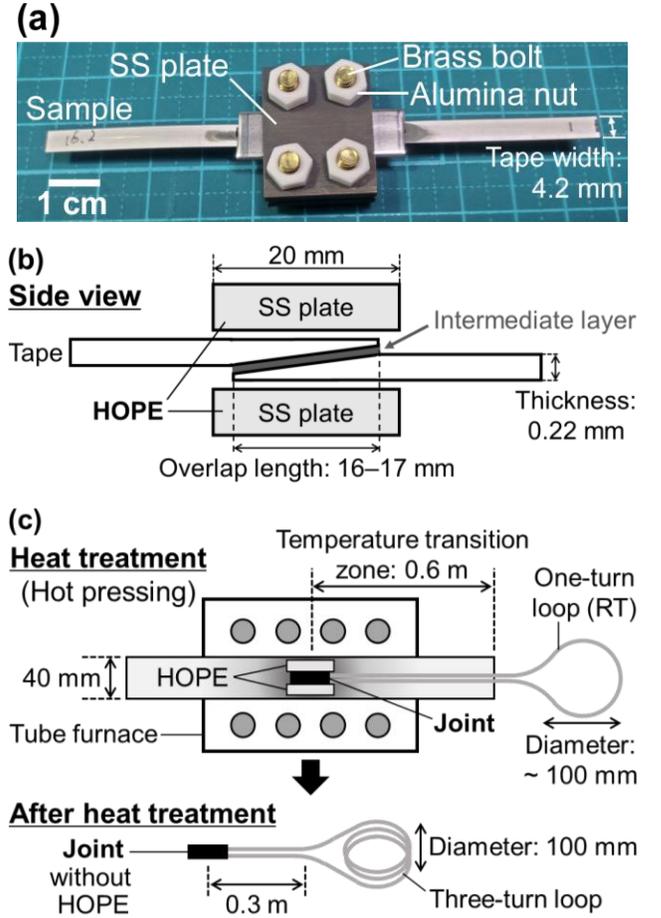


Fig. 1. (a) Photograph of straight joint sample before heat treatment with HOPE (HOt-Pressing Equipment). (b) Schematic of side view of straight joint sample with HOPE. (c) Schematic of fabrication procedure for closed-loop sample CL-02.

for the joint, CL-02 was a one-turn loop with the about 0.6 m long temperature transition zone. The joint was inserted in the tube furnace and heat-treated, while the loop part was outside the furnace and held at room temperature [4]. After the heat treatment and removal of HOPE, as shown in the lower part of Fig 1(c), the one-turn loop was wound into three-turn loop with a diameter of 100 mm. This procedure did not damage the tape and joint. This sample geometry is suitable for the evaluation system described in the next section.

In general, because the Bi-2223 formation reaction is slow, a heat treatment of more than 24 h is necessary to ensure high purity when the starting material is precursor powder [25]. To synthesize a high-purity Bi-2223 intermediate layer by a short heat treatment, we premixed Bi-2223 powder with a slurry.

Figs. 2 and 3 show the flow chart for the powder and slurry preparation processes and the $\theta/2\theta$ x-ray diffraction patterns for the three types of powder measured using a Rigaku Mini-Flex II, respectively. The precursor powder produced by TEP Co., Ltd. was used as the starting material. The nominal chemical composition of the precursor powder was Bi:Pb:Sr:Ca:Cu = 1.6:0.45:1.9:2.0:3.0. The main phase of the precursor pow-

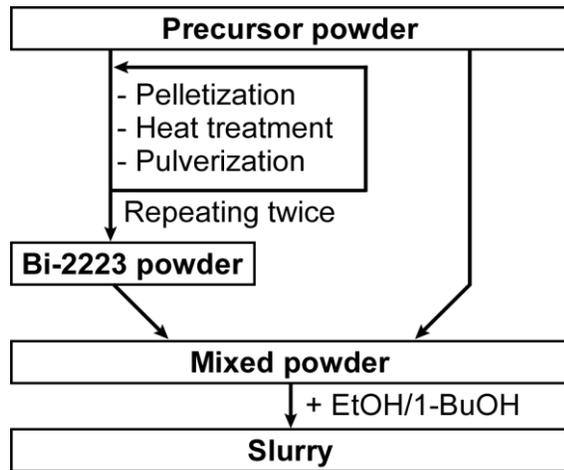


Fig. 2. Powder and slurry preparation flow chart.

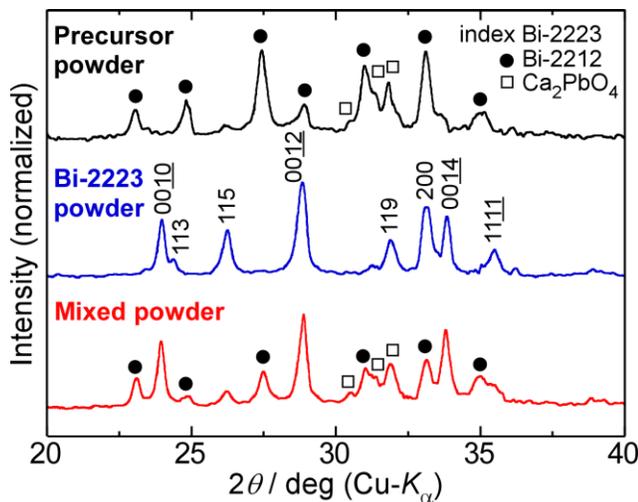


Fig. 3. $\theta/2\theta$ x-ray diffraction patterns of three types of powder used in this study.

der was $(\text{Bi,Pb})_2\text{Sr}_2\text{CaCu}_2\text{O}_y$ (Bi-2212). To synthesize the Bi-2223 powder, the precursor powder was pelletized, heat-treated at a P_{O_2} of 3 kPa and 820 °C, and pulverized. After repeating twice these steps, the Bi-2223 powder was obtained. By using the two types of powder (i.e., the precursor powder and Bi-2223 powder), we obtained a mixed powder. A slurry was prepared by adding EtOH/1-BuOH as solvent to the mixed powder.

The heat treatment profile used during the hot-pressing process is shown in Fig. 4. The heat treatment at a P_{O_2} of 3 kPa in a tube furnace was completed in about 24 h. To avoid overshooting the holding temperature, a slow heating rate was used. In addition, a slow cooling rate, which is known to be effective in increasing the I_c of Bi-2223 tapes [26], was also used.

The pressure was applied on the joint using HOPE during the heat treatment. The applied pressures (P) for the straight joint samples SJ-02 and SJ-05 at RT were about 5 and 13 MPa ($N = 0.20$ and 0.50 N·m), respectively. The pressure values were checked using pressure measurement films (Fujifilm Prescale). For comparison, SJ-0 was prepared without applying

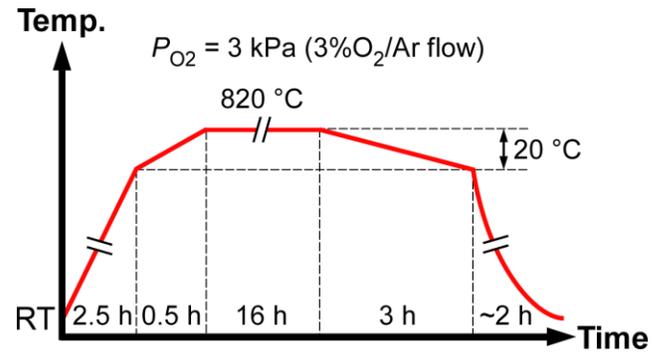


Fig. 4. Heat treatment profile used for all samples in this study. Heat treatment was completed in about 24 h.

TABLE I
SPECIFICATIONS OF STRAIGHT JOINT SAMPLES. TRANSPORT MEASUREMENTS IN SELF-FIELD WERE PERFORMED IN LIQUID NITROGEN BATH

Sample	Bolt-tightening torque, $N/N\cdot m$	Applied pressure to joint at RT, P/MPa	Transport measurements (77 K)		
			Before or after removal of HOPE	I_c/A	$n/-$
SJ-0	0 ¹⁾	0 ¹⁾	–	0.34	1.89
SJ-02	0.20	~5	Before	57.8	12.4
			After	40.5	12.4
SJ-05	0.50	~13	Before	36.3	4.64
			After	34.6	4.60

¹⁾SJ-0 was prepared without applying the pressure during the heat treatment.

any pressure ($P = 0$) during the heat treatment. The heat treatment profile of SJ-0 was the same as that of the hot-pressed samples. The specifications of the straight joint samples are listed in Table I. P was about 5 MPa ($N = 0.20$ N·m) for the joint in the closed-loop sample CL-02.

C. Characterization

Transport measurements in self-field were performed for the three straight joint samples in a liquid nitrogen bath (77 K) using the conventional DC four-probe method. For the hot-pressed samples SJ-02 and SJ-05, the transport measurements were performed twice. First, the voltage–current (V – I) curve was obtained for the samples while HOPE was attached. Subsequently, HOPE was removed by loosening the bolts, resulting in the release of the pressure applied by HOPE. The V – I curve was measured again using the samples without HOPE. I_c was determined using a $0.2 \mu\text{V}$ voltage criterion. The exponent, n , of the empirical power law model was calculated for the voltage range of $0.2 \mu\text{V} \leq V \leq 0.7 \mu\text{V}$.

After the transport measurements, we observed the microstructures of the straight joint samples. Note that HOPE had already been removed from the hot-pressed samples at this stage. The polished surfaces of the transverse cross-sections of the three straight joint samples were observed. Secondary and backscattered electron images were obtained using a field emission scanning electron microscope (FE-SEM, Hitachi SU-70). The local chemical composition was analyzed using an

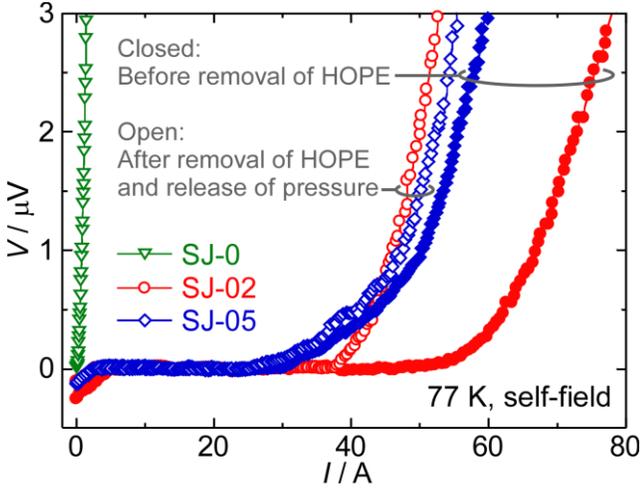


Fig. 5. V - I curves for three straight joint samples at 77 K in self-field. SJ-0 was not superconductive. In hot-pressed samples SJ-02 and SJ-05, superconducting joints were formed.

energy dispersive x-ray spectroscopy (EDS) system attached to the FE-SEM.

Current decay method is known to evaluate an R_j value of $10^{-13} \Omega$ or lower, which cannot be evaluated by transport measurements [1]. Current decay measurements at 4.2 K in self-field were performed for the closed-loop sample CL-02 using the joint resistance evaluation system that we have developed [27]. CL-02 was mounted on the sample holder after the removal of HOPE, because the space for the joint within the holder was less than 25 mm in diameter. After cooling to 4.2 K, loop current (I_{loop}) was injected in CL-02 by magnetic induction using a copper coil located at the loop center. Time dependence of I_{loop} was measured using a current transformer [28].

III. RESULTS AND DISCUSSION

A. Transport measurements of straight joint samples at 77 K in self-field

Fig. 5 shows the V - I curves at 77 K in self-field for the three straight joint samples. SJ-0 showed an almost linear increase in voltage, which meant that SJ-0 was not superconductive. This is attributed to the poor grain connectivity and low intergrain J_c of the intermediate layer. It is probably due to the short heat treatment (24 h) and the use of the Bi-2223-rich powder for synthesizing the intermediate layer [29].

The V - I curves for SJ-02 and SJ-05 showed a typical superconducting to normal transition. This indicates that Bi-2223 superconducting joints were successfully formed in these hot-pressed samples. It also suggests that hot pressing is effective for improving the grain connectivity and synthesizing a high- J_c intermediate layer.

Before the removal of HOPE, SJ-02 showed an I_c of 57.8 A. Although this I_c value is not much higher than those of the previously reported joint samples, all the previous samples

were fabricated with longer processing times than SJ-02 [3], [4], [17]. Thus, hot pressing is effective for fabricating Bi-2223 superconducting joints with practical I_c values even by the short heat treatment (24 h). However, considering that the I_c of a virgin tape is more than 150 A at 77 K in self-field, an I_c value of the hot-pressed superconducting joints has room for improvement.

After the removal of HOPE, that is, after the release of the pressure, the I_c of SJ-02 decreased by 30%, although the n value did not change. We observed an increase in the thickness of the joint of about 20 μm after the removal of HOPE. Assuming that this increase was mainly attributable to the expansion of the intermediate layer, the increase corresponded to the decrease in the density of the intermediate layer with microstructural changes, which, in turn, resulted in a decrease in the intergrain J_c . This is a possible reason for the decrease in the I_c of SJ-02 after the removal of HOPE. This decrease in the I_c is not reversible. In preliminary experiments, we found that the I_c was not recovered and further decreased by reapplying HOPE to a joint sample.

We expected that a higher P would result in the densification of the intermediate layer and an increase in I_c . However, SJ-05 exhibited the lower I_c and n values than those of SJ-02. The P value for SJ-05 was probably too high and mechanically damaged the Bi-2223 filaments in the tapes during the heat treatment. This result is similar to the low I_c and n values obtained in the case of the samples pressed at high pressures and RT in our previous study [17].

For SJ-05, the increase in the joint thickness after the removal of HOPE was about 70 μm . This suggests that in the intermediate layer, both the decrease in the density after the removal of HOPE and the densification effect by hot pressing should have been larger in the case of SJ-05 compared with those for SJ-02. However, the I_c and n values of SJ-05 were lower than those of SJ-02 before the removal of HOPE and decreased to a lesser extent after the removal of HOPE. This implies that the I_c and n values of SJ-05 are determined by the superconducting properties of the damaged Bi-2223 filaments in the tapes.

B. Microstructural observations of straight joint samples

Fig. 6 shows typical secondary electron images of the polished surfaces of the transverse cross-section of the joining part of the samples. The upper and lower parts correspond to the intermediate layer and the Bi-2223 tape, respectively.

Many voids were observed in the intermediate layer of each sample. In the case of the hot-pressed samples, although the microstructural observations were performed after the transport measurements, that is, after the removal of HOPE, the density of the intermediate layer appeared to be higher than that of SJ-0. The filling factor values of the intermediate layer evaluated by image analysis [17] were about 70% for SJ-0 and about 80% for both of SJ-02 and SJ-05.

In the case of SJ-02 and SJ-05, the grains were in good contact at the interface between the intermediate layer and filaments of the tape. This microstructure contributed to the for-

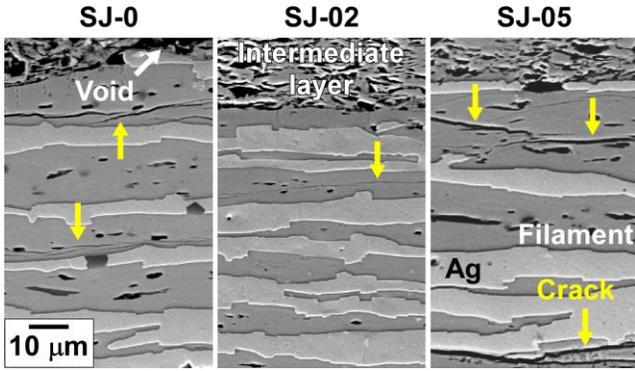


Fig. 6. Typical secondary electron images of polished surfaces of transverse cross-sections of joining part of straight joint samples. Microstructural observations of hot-pressed samples were performed after removal of HOPE. In SJ-0, many voids at joining interface were observed. Some large cracks were observed only in Bi-2223 filaments of SJ-05.

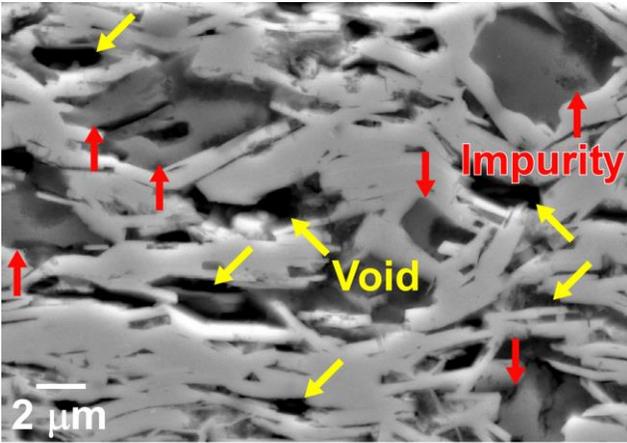


Fig. 7. Backscattered electron image of intermediate layer of SJ-02. Impurities 2–5 μm in size (Ca_2CuO_3) remained probably owing to short heat treatment (24 h).

mation of the superconducting joints in the hot-pressed samples. In contrast, many voids were observed at the joining interface in SJ-0. This poor grain connectivity at the interface is one of the reasons why SJ-0 was not superconductive. These results imply that hot pressing is effective not only for densifying the intermediate layer but also for forming a joining interface wherein the grains are in good contact.

Large cracks were observed in the Bi-2223 filaments of SJ-05. In contrast, such cracks were not observed in SJ-0 and SJ-02. These large cracks represent mechanical damage to the filaments. This suggests that the superconducting properties of the damaged filaments had a determining effect on the I_c and n values of SJ-05.

The microstructural observations also revealed that the purity of the intermediate layer was insufficiently high. Fig. 7 shows a backscattered electron image of the intermediate layer of SJ-02. Voids (black region), plate-like Bi-2223 grains (light gray region), and 2–5 μm sized impurities (dark gray region) were observed. Based on the analyses of the local chemical composition, the secondary phase was determined to be Ca_2CuO_3 . The impurities remained probably owing to the

short heat treatment (24 h), because Ca_2CuO_3 is the precursor to the formation of the Bi-2223 phase [30], [31]. Such impurities reduce the intergrain J_c of the intermediate layer, which, in turn, decreases the I_c of the superconducting joints.

C. Evaluation of R_j and persistent current using closed-loop sample with a superconducting joint

Fig. 8 shows the time dependence of I_{loop} at 4.2 K in self-field for the closed-loop sample CL-02 obtained by the current decay measurements. I_{loop} was injected at $t = 0$. An initial decrease in I_{loop} of about 0.02 A was observed for 1.5×10^3 s. Thereafter, the time variation in I_{loop} gradually became smaller. This result is similar to those of the previous current decay measurements [4], [27], [28] and indicates that persistent current of about 130 A was flowing in CL-02.

Time dependence of I_{loop} can be fitted using an exponential decay function [1]. Assuming that the resistance of the sample corresponds to that of the superconducting joint, the decay time constant is L/R_j . Based on the fitting of the experimental data points between 4×10^3 and 5×10^3 s, the R_j at 4.2 K in self-field was deduced to be $7.3 \times 10^{-15} \Omega$, as shown in Fig. 8 (solid line). This value is comparable to that observed for a previous Bi-2223 closed-loop sample with a superconducting joint [4]. It is also comparable to or lower than that of REBCO [28], [32] and LTS [1], [2], [27] superconducting joints. From these results, it can be concluded that the superconducting joint was successfully formed in CL-02 by the hot-pressing process.

After the current decay measurements, the temperature of CL-02 was started to increase while I_{loop} of about 130 A was still flowing. The temperature was increased from 4.2 to 120 K at a ramp rate of 0.5 K min^{-1} using a heater installed in the sample holder [27]. During this temperature increase, we measured the temperature of superconducting joint (T_j) of CL-02. Fig. 9 shows T_j dependence of I_{loop} in self-field. I_{loop} was constant till $T_j = 22 \text{ K}$. When T_j was increased beyond 22 K, I_{loop} decreased with increasing T_j . I_{loop} disappeared completely at $T_j = 103 \text{ K}$, as shown in inset of Fig. 9. This means that the critical temperature (T_c) of the joint in self-field was 103 K. This value is almost the same as that of a Bi-2223 superconducting joint sample evaluated by transport measurements in our previous study [3].

At T_j values of above 30 K, a linear relationship was observed between T_j and I_{loop} . In this region, I_{loop} corresponded to the maximum value of the persistent current that can flow in CL-02. At a given temperature, the maximum value of the persistent current would correspond to I_c , which was determined by a very low-voltage criterion such as less than 10^{-8} V , of the superconducting joint. At T_j of around 70 K, the slope of the $I_{\text{loop}}-T_j$ curve appears to change. Although we observed similar slope changes in preliminary experiments using other Bi-2223 closed-loop samples, the reason of this change is not clear.

The gray dashed line in Fig. 9 was derived from the data points between T_j values of 30 and 60 K using the least-squares method. In transport measurements using Bi-2223 su-

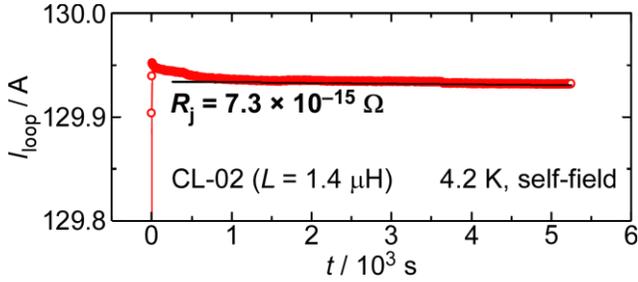


Fig. 8. Time dependence of I_{loop} for CL-02 at 4.2 K in self-field. Solid line represents exponential decay curve with $R_j = 7.3 \times 10^{-15} \Omega$, which was deduced from fitting of experimental data points between 4×10^3 and 5×10^3 s. It was indicated that persistent current was flowing in CL-02.

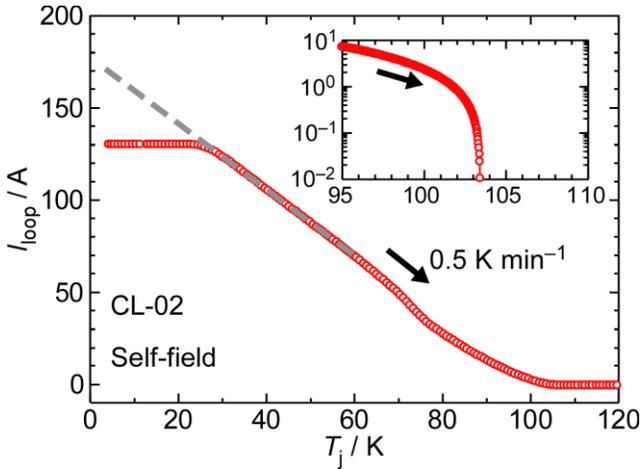


Fig. 9. T_j dependence of I_{loop} for CL-02 in self-field to evaluate maximum value of persistent current that can flow in CL-02 at the given temperature. Inset shows T_c of joint was about 103 K. Gray dashed line was derived from data points between T_j values of 30 and 60 K using least-squares method. Maximum value of persistent current was estimated to be about 170 A at 4.2 K.

perconducting joint samples, a linear relationship between I_c and T_j was observed at 4.2–90 K [3]. Assuming that the similar linear relationship holds in the $I_{\text{loop}}-T_j$ curve even at low temperatures, the maximum value of the persistent current that can flow in CL-02 is estimated to be about 170 A at 4.2 K. Note that this value was much lower than I_c of a virgin tape (more than 800 A in magnetic fields below 1 T [12]).

D. Yields, processing time, and issues to be addressed

In this study, we demonstrated the Bi-2223 superconducting joints using the hot-pressing process. Although not mentioned here, we have fabricated more than 20 hot-pressed joint samples with the P of about 1–13 MPa in preliminary experiments and confirmed that superconducting joints were formed in each case. In contrast, as demonstrated in SJ-0, non-superconductive joint samples have sometimes been fabricated without applying a hot-pressing pressure. We believe that the superconducting joints can be fabricated in high yields by the hot-pressing process.

The heat treatment to fabricate the hot-pressed superconducting joints was completed within about 24 h. This short

heat treatment reduced the processing time for joining by more than a day compared with that for previous studies [3], [4], [17]. Note that the use of a more precisely controlled furnace can further reduce the heat treatment time by allowing for higher heating and cooling rates.

Even before the removal of HOPE, the hot-pressed superconducting joints showed lower I_c than a virgin tape. A few issues related to the hot-pressing process need to be addressed to increase I_c . First, the purity of the intermediate layer should be improved. Reducing the impurities in the intermediate layer will be effective for increasing the intergrain J_c , as demonstrated in the case of Bi-2223 tapes [13]. A long heat treatment will reduce Ca_2CuO_3 with the formation of Bi-2223 [30]. Although the processing time is extended, a long heat treatment probably leads to an increase in the I_c of the superconducting joints.

Second, the I_c of the hot-pressed superconducting joints can be further increased by densifying the intermediate layer. We will investigate the upper limit for densification by hot pressing without damaging the Bi-2223 filaments in the tapes. We will also attempt to introduce the two-step heat treatment process, which is effective for the densification [17].

In preliminary experiments using the closed-loop sample CL-02, we observed the persistent current of about 60 A at 4.2 K with applying 1 T to the joint. Considering a large decrease of the I_c in a magnetic field similar to the previous study [4], the in-field I_c of the hot-pressed superconducting joints is also required to be increased. Details of the in-field performance will be reported elsewhere.

IV. SUMMARY

Bi-2223 superconducting joints were successfully formed in samples fabricated by the hot-pressing process with a 24 h heat treatment. An I_c of 57.8 A at 77 K in self-field under a 0.2 μV voltage criterion was observed in the hot-pressed sample. The superconducting joint in the closed-loop sample showed an R_j of $7.3 \times 10^{-15} \Omega$ at 4.2 K in self-field. It was indicated that a persistent current was flowing in the loop. The maximum value of the persistent current that could flow in the closed-loop sample was estimated to be about 170 A at 4.2 K.

Issues to be addressed are purification and further densification of the intermediate layer, which is probably effective for improving the I_c , including in magnetic fields, of the hot-pressed superconducting joints. Once these issues are addressed, the proposed hot-pressing process should be a promising method to fabricate high-performance Bi-2223 superconducting joints in high yields and within a short processing time.

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