Development of a Persistent Current Mode 9.39 T (400 MHz) LTS/Bi-2223 NMR Magnet with a Bi-2223 Superconducting Joint

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***Abstract*—We developed and operated a persistent current (PC) mode 9.39 T (400 MHz for ^{1}H NMR) LTS/Bi-2223 NMR magnet using a Bi-2223 insert coil with a superconducting joint. Firstly, we developed a basic joining procedure to achieve the superconducting joint for the high-strength Bi-2223 tape used for the insert coil. We applied this procedure to the coil. Secondly, a stand-alone PC mode operation test for the coil at 4.2 K was performed. No apparent field decay was observed owing to the superconducting joint. Finally, we installed the coil inside the LTS outer coil's bore to form a PC mode LTS/Bi-2223 NMR magnet system. We successfully demonstrated a 9.39 T PC mode operation of the magnet though the current of the insert coil was limited to 5 A. The field drift rate was as low as 0.4×10^{-3} ppm h^{-1}. The joint resistance was estimated to be $< 10^{-12}$ Ω at 4.2 K and 0.26 T. To our best knowledge, this is the first demonstration of a Bi-2223 superconducting joint that realized a PC mode LTS/Bi-2223 NMR magnet. This is an important step towards full-scale PC mode Bi-2223 magnets, including a 30.5 T (1.3 GHz) LTS/HTS NMR magnet.**

***Index Terms*—Superconducting Magnets, Bi-2223 Tape, Nuclear magnetic resonance**

I. Introduction

UNDER the JST-Mirai Program, we have been developing a PC mode 30.5 T (1.3 GHz) nuclear magnetic resonance (NMR) magnet using low-temperature superconductor (LTS) outer coils and high-temperature superconductor (HTS) inner/middle coils [1][2][3]. The magnet comprises a REBa$_2$Cu$_3$O$_y$ (REBCO, RE = Rare earth) inner coil, a (Bi,Pb)$_2$Sr$_2$Ca$_2$Cu$_3$O$_y$ (Bi-2223) middle coil, and LTS outer coils. All the coils are to be connected in series. The quantitative requirement for the joints between HTS conductors is an ultra-low resistance of $< 10^{-12}$ Ω per joint at the operating current of 231 A in an external field of < 1 T at 4.2 K. Practically, such a low resistance should be provided by the superconducting joint [4]. To develop practical HTS superconducting joint technologies and to implement joints in the HTS coils are the significant issues to be addressed from the viewpoint of achieving a PC mode operation. In this study, we developed a PC mode 9.39 T (400 MHz) LTS/Bi-2223 NMR magnet using a Bi-2223 insert coil with a superconducting joint. This development is a good partial demonstration of the 30.5 T magnet and provides insight into the technological issues.

In a previous work [5], Takeda et al. reported a superconducting joint technology providing high critical current (I_c) characteristics between commercially available silver-sheathed multifilamentary Bi-2223 tapes without mechanical reinforcement (DI-BSCCO® Type H [6]). In a more recent study [7], an improved I_c of ~ 300 A at 4.2 K and 1 T under a 10^{-9} Ω criterion was obtained for a joint with praying-hands configuration, which is geometrically required to be installed in a coil. Furthermore, a low joint resistance of $< 10^{-14}$ Ω at 4.2 K, 1 T, and ~ 140 A was observed in a five-turn loop sample with the superconducting joint. However, these studies did not use the high-strength Bi-2223 tape laminated with Ni-alloy reinforcing tapes (DI-BSCCO® Type HT-NX [8]). The high-strength tape is indispensable for a practical coil under a high hoop stress, including the middle coil of the 30.5 T magnet.

Firstly, we developed a basic joining procedure for the high-strength Bi-2223 tape. We applied this procedure to the insert coil wound with a single piece of the high-strength tape. Secondly, we performed a stand-alone PC mode operation test for the coil at 4.2 K. Finally, we constructed an LTS/Bi-2223 NMR magnet. The magnet was operated in a 9.39 T PC mode at 4.2 K. We measured the temporal drift of the magnetic field and estimated the Bi-2223 joint resistance from the drift rate.

II. Magnet Specification

Design parameters for the PC mode 9.39 T LTS/Bi-2223 NMR magnet are shown in Table I. The NMR magnet had a

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54 mm room temperature bore. The LTS outer coils were re-used from a 9.39 T PC mode LTS/REBCO NMR magnet [9]

Figures 1(a) and 1(b) show a schematic of cross-section of the magnet and a photograph of the Bi-2223 insert coil with a fiber-reinforced plastic (FRP) top plate, respectively. The Bi-2223 coil was wound by layer-winding and both ends of the coil were guided from the winding pack to terminal wiring on the FRP top plate by spiraling up the SUS tube guide. While the winding pack and spiral on the SUS tube guide were wax impregnated, the terminal wiring on the plate was not impregnated and only reinforced by being sandwiched between a pair of SUS tapes. Therefore, there was slight room for the terminal wiring to be moved. The terminal wiring included a joint and persistent current switch (PCS) as described later.

Figure 1(c) shows a schematic circuit of the magnet. The insert coil with a Bi-2223 PCS and the outer coils with a NbTi PCS were operated in the individual PC circuit. Two power supplies were used for charging the magnet. The coils were cooled at 4.2 K in liquid helium. The magnet installed a 4 K pulse-tube cryocooler for zero boil off of liquid helium.

A Bi-2223 PCS function was manufactured on one of the terminal conductors of the coil. A schematic of the PCS function is shown in Figure 1(d). We removed the Ni-alloy reinforcing tapes over ~ 5 cm by a heating method which can avoid buckling of a Bi-2223 tape [10]. A ϕ0.1 mm nichrome heater was wound on this part. The PCS part was embedded in epoxy resin (Araldite® AV/HV 1580) for thermal insulation. The removal of the reinforcing tapes and embedment in epoxy resin were effective to reduce a heater power to obtain an off-resistance. The off-resistance of the PCS of this coil in liquid helium arose at a heater power of 9.6 W. The estimated off-resistance was ~ 0.7 mΩ based on an extrapolation from the resistance measurements in liquid nitrogen. This Bi-2223 PCS function has been separately developed and qualified, which will be reported elsewhere [11].

III. Superconducting Joints between High-Strength Bi-2223 Tapes

We developed the basic joining procedure applicable for the high-strength Bi-2223 tapes. We removed the reinforcing materials including the bonding solder. The Ni-alloy reinforcing tapes were removed by the heating method [10]. We then removed the solder, which had been remaining on the surfaces of the Bi-2223 tapes, by chemical etching with an acid reagent. After removing the solder, a clean and flat silver surface of the Bi-2223 tape was obtained, which was confirmed by x-ray diffraction measurements and microstructural observation. We also confirmed that I_c degradation at 77 K in self-field was sufficiently small through the removal process.

After this process, the joining processes [5][7] were applied to form a superconducting joint. The joining condition is summarized in Table II. The removal process was applied to a pair of the ~ 15-cm-long high-strength tapes to fabricate a short sample test joint in the praying-hands configuration. After the short sample test, we made the praying-hands-type joint between the terminal conductors of the Bi-2223 insert coil, i.e., coil joint, through the removal process over a ~ 30-cm-long region for each terminal.

We made current transport measurements in liquid nitrogen for the short sample joint and coil joint at 77 K in self-field.

TABLE I
Design Parameters of a Bi-2223 Insert Coil and LTS Outer Coils for a PC Mode 9.39 T (400 MHz) LTS/Bi-2223 NMR Magnet.

	Unit	Bi-2223 inner coil	LTS outer coils
Coil Conductor	-	DI-BSCCO® Type HT-NX	Nb_3Sn, NbTi
Conductor width: thickness	mm	4.5: 0.3	-
Conductor I_c (77 K, self-field)	A	~ 200	-
Conductor length	m	196	-
ID: OD: Height	mm	99.7:105.3:345	133:332:547
Total turns	-	608	-
Number of layers	-	8	-
Coil constant	mT A^{-1}	2.09	69.02
Self-inductance	H	0.0104	70.0
Mutual inductance	H	0.34	
PCS conductor	-	DI-BSCCO® Type HT-NX[1)]	NbTi/CuNi
400 MHz operation at 4.2 K			
Operating current	A	10.0	135.90
Magnetic field	T: MHz	0.021: 0.90	9.379: 399.32
Local field intensity at the joint	T	0.26	-

[1)] Schematic of the Bi-2223 PCS function is shown in Figure 1(d)

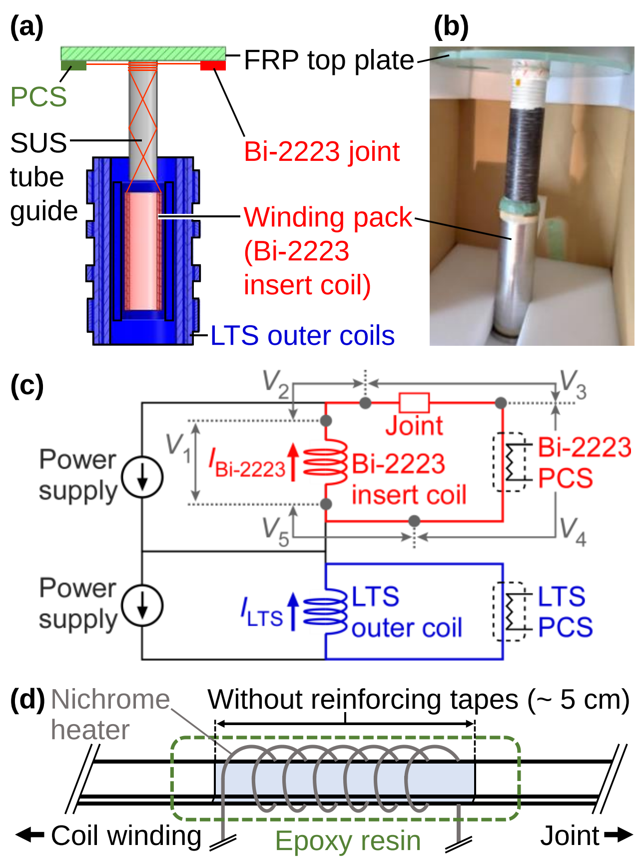

Figure 1 (a) Schematic of cross-section of a PC mode 9.39 T LTS/Bi-2223 NMR magnet. (b) Photograph of the Bi-2223 insert coil with a FRP top plate to locate the terminal wiring of the coil. (c) Schematic circuit of the NMR magnet. V_1, V_2, V_3, V_4, and V_5 represent voltage in each section of the insert coil. (d) Schematic of the Bi-2223 PCS function of the insert coil.

TABLE II
JOINING CONDITION FOR A SHORT SAMPLE TEST JOINT AND COIL JOINT

	Unit	Condition
Uniaxial pressing	MPa	~ 200
Heat treatment	-	810°C, 24 h, P_{O2} = 3 kPa[2]
Area of the intermediate layer	mm^2	~ 70
Number of the exposed filaments	-	~ 90 (70–80% of the total number of filaments)

[2] Partial oxygen pressure (P_{O2}) was controlled by flowing a 3%O_2/Ar gas.

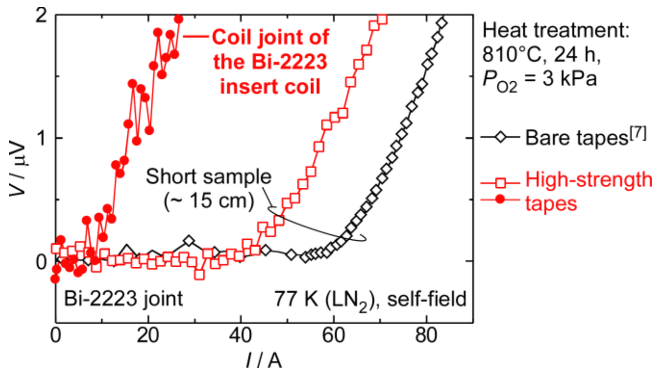

Figure 2 *V-I* curves for joints with praying hands configuration at 77 K in self-field. A *V-I* curve for a joint between the bare tapes (DI-BSCCO® Type H) [7] is also shown.

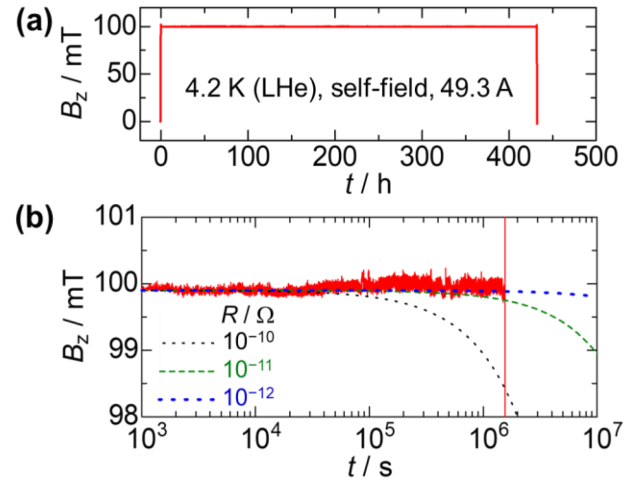

Figure 3 The magnetic field (B_z) versus time in the stand-alone PC mode operation test for the Bi-2223 insert coil in liquid helium at 4.2 K and 49.3 A. (a) Overview (b) Magnified view with exponential decay curves corresponding to $R = 10^{-12}$–10^{-10} Ω.

Figure 2 shows the obtained *V–I* curves. For comparison, the figure also displays the *V–I* curve of a short sample joint between bare Bi-2223 tapes (DI-BSCCO® Type H) fabricated by the same condition [7]. Each joint exhibited the general superconducting-to-normal transition under the accuracy of the voltage measurements. These results imply that the superconducting joint was formed in each joint.

However, the transition current values were different. The short samples for the high-strength tapes and bare tapes showed voltage rises at ~ 40 A and ~ 60 A, respectively. This indicates that the joining condition for the high-strength tapes should be optimized. Conversely, it is obvious that the transition current of the coil joint, ~ 10 A, is much lower than those of the short samples.

There were difficulties peculiar to the fabrication of the coil joint, which probably influenced the current transport properties. We had to handle a pair of the long terminal conductors of ~ 1 m in length without degradation. This is because we heat-treated the joint in a tube furnace while the winding pack and spiral on the SUS tube guide held at room temperature. Furthermore, the ~ 30-cm-long end part of the terminal conductors was brittle since the reinforcing materials were removed.

IV. A STAND-ALONE TEST FOR THE Bi-2223 INSERT COIL

Before the construction of the whole LTS/Bi-2223 NMR magnet, we performed a stand-alone PC mode operation test for the insert coil at 4.2 K. The coil was installed in an open dewar and cooled with liquid helium. Figure 3(a) shows the overview of the magnetic field (B_z) versus time measured with a Hall censor installed in the coil center. We charged the coil to 49.3 A via 50.2 A, i.e., a 1.8% current sweep reversal to suppress a temporal drift of the field due to the relaxation of screening currents [9][12]. We started the PC mode operation at t = 0 and discharged the coil at t = 432 h. During the test, no apparent field decay was observed within the accuracy of the Hall sensor.

Figure 3(b) shows the magnified view of B_z–t curve. Exponential decay curves are also displayed, each of which corresponds to a certain resistance of the coil circuit (R) of 10^{-12}–10^{-10} Ω with a self-inductance of 10.4 mH. The value of R is affected by several factors other than the joint resistance, such as the relaxation of screening currents. However, we can estimate the joint resistance of $< 10^{-11}$ Ω. The test results indicate that the PC circuit is successfully formed in the coil owing to the superconducting joint.

V. A 9.39 T PC MODE OPERATION TEST FOR THE LTS/Bi-2223 NMR MAGNET

From the stand-alone test results, it was indicated that the Bi-2223 insert coil can operate in the PC mode as a part of the NMR magnet. We installed the coil inside the LTS outer coils' bore and constructed the whole NMR magnet system.

Before charging the outer coils, we performed a self-field PC mode operation test for the insert coil to check the performance of the insert coil. Firstly, we charged the insert coil to 50 A under the PCS off-state. A resistive voltage of ~ 4 μV at 50 A appeared on the coil circuit of V_1 seen in Figure 1(c), which was not observed in the stand-alone test. V_1 included the winding pack, the spiral on the SUS tube guide, and part of the terminal wiring on the FRP top plate. Secondly, the power supply current was reduced under the PCS on-state. In this process, the current for the joint and PCS increased from 0 A to 50 A. No resistive voltage was observed in V_2, V_3 (joint), V_4 (PCS), and V_5 seen in Figure 1(c). Thirdly, in the following PC mode operation, we observed a notable field decay. After the substantial decay, the field became stable at a certain value corresponding to the coil current of ~ 26 A. Finally, we discharged the insert coil.

These results indicate that there was degradation in the current transport properties for the insert coil within the coil circuit of V_1. No evidence of degradation was found for the other part of the circuit including the joint and PCS in the current range of < 50 A. The reason of the degradation has not been clear thus far. The degradation should have occurred in the processes after the stand-alone test. The processes included ground transportation between Yokohama and Kobe, construction of the LTS/Bi-2223 integrated coil magnet, and thermal cycling between room temperature and 4.2 K. During these processes, there was no mechanical handling of the terminal wiring.

As described above, the insert coil circuit maintained its superconductivity although it had degradation. There was a possibility that the LTS/Bi-2223 NMR magnet would be able to operate in the PC mode when the insert coil current was sufficiently low. Based on this assumption, we performed the PC mode operation test of the NMR magnet.

Firstly, we charged the outer coils to 135.9 A via 136.2 A, i.e., a 0.22% current sweep reversal. Under the PC mode operation for the outer coils, a stable field with a small drift rate of 7×10^{-3} ppm h^{-1} was observed ~ 15 h after charging. Secondly, we charged the insert coil to the design operating current of 10.0 A and started the PC mode operation. However, the magnetic field started to decay notably; this was because the current transport properties of the degraded part reduced due to the external field by the outer coils. With a logarithmic decay trend of the field, we estimated that the PC mode operation at ~ 5.2 A in the insert coil was feasible. Thus, we decreased the insert coil current to 5.0 A via 4.0 A with applying the current sweep reversal method. Eventually, a 9.39 T (400 MHz) PC mode operation for the LTS/Bi-2223 NMR magnet started. The field of the insert coil was 0.011 T (0.45 MHz).

The magnetic field was measured with an NMR Teslameter (PT2025, Metrolab Technology SA). Figure 4 shows the magnetic field drift in the PC mode operation for the NMR magnet. Large fluctuations of the field were due to the external effects such as a fluctuation of the inside pressure caused by liquid helium refill. From the measured data between $t = 8 \times 10^2$ h and 2×10^3 h, the field drift rate was calculated to be 0.4×10^{-3} ppm h^{-1}. This value was much lower than the requirement for the solution NMR measurements (< 0.01 ppm h^{-1}).

Considering the magnetic coupling of the Bi-2223 insert coil and the LTS outer coils, the Bi-2223 joint resistance was estimated [9]. We assumed that the resistance of the LTS outer coil circuit was 2×10^{-10} Ω, which corresponded to a standard field drift rate of 0.01 ppm h^{-1}. We ignored the effect of the relaxation of the screening currents for the insert coil under the assumption that the current sweep reversal suppressed the relaxation. The resistance of the Bi-2223 insert coil circuit was estimated to be 5×10^{-13} Ω. We cannot deny a possibility that the resistance was due to the degraded part of the insert coil. However, we can estimate the Bi-2223 joint resistance to be $< 10^{-12}$ Ω at 4.2 K and 5.0 A in an external field of 0.26 T.

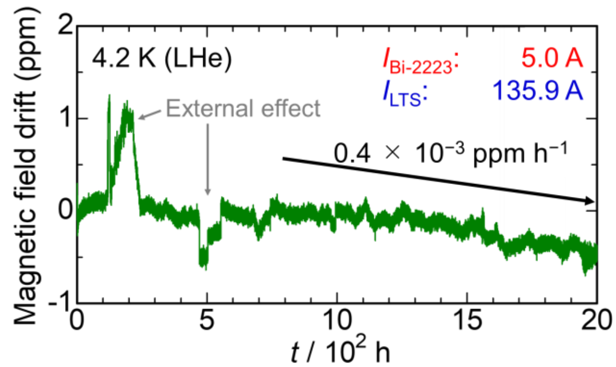

Figure 4 Magnetic field drift for the LTS/Bi-2223 NMR magnet in the PC mode operation. The field was measured with an NMR Teslameter (PT2025, Metrolab Technology SA).

VI. Discussion and Future Prospects

To our best knowledge, the present work is the first demonstration of the Bi-2223 superconducting joint that realized a PC mode LTS/Bi-2223 NMR magnet. The estimated joint resistance satisfies one of the quantitative design requirements for the PC mode 30.5 T LTS/HTS NMR magnet ($< 10^{-12}$ Ω) [1] though the operating current was limited. This provides a hopeful perspective for the realization of full-scale PC mode Bi-2223 magnets including the 30.5 T magnet.

For such magnets, the PC mode operation of the Bi-2223 coils showing a sufficiently low resistance has to be demonstrated at a high operating current such as > 200 A. The issues have remained to be addressed to achieve this. Firstly, a joining procedure to realize a coil joint showing high I_c has to be established. We have aimed to establish a strategy to achieve high I_c for a short joint [7]. We have recently found that I_c of a short sample for the high-strength tapes can be enhanced up to the comparable level of that for the bare tapes by modifying the heat treatment process [13]. In addition, we plan to work on technology to apply the strategy to a coil joint. We will design a method to overcome the difficulties peculiar to the fabrication of a coil joint described above. This will lead to establishment of a joining procedure to realize a coil joint showing sufficiently high I_c.

Secondly, we must establish a mechanical structure and handling method of a Bi-2223 coil with a particular focus on terminal wiring to prevent degradation of the coil performance. We plan to examine where the degradation occurred in the insert coil after the operation for the NMR magnet is finished. Effects of thermal cycling and the mechanical stress on the coil performance will be carefully evaluated.

VII. Summary

(1) Towards a PC mode 30.5 T (1.3 GHz) LTS/HTS NMR magnet, we developed the PC mode 9.39 T (400 MHz) LTS/Bi-2223 NMR magnet using the Bi-2223 insert coil.
(2) We developed a basic joining procedure for the high-strength Bi-2223 tape used for the insert coil. The super-

conducting joint was realized between the terminal conductors of the coil.

(3) We made a stand-alone PC mode operation test for the coil in liquid helium at 4.2 K and 49.3 A. No apparent field decay was observed.

(4) A 9.39 T PC mode operation for the LTS/Bi-2223 NMR magnet was successfully demonstrated at 4.2 K. The field drift rate was as low as 0.4×10^{-3} ppm h^{-1}. The Bi-2223 joint resistance was estimated to be $< 10^{-12}\ \Omega$ at 5.0 A in an external field of 0.26 T. Although the operating current was limited, to our best knowledge, the present work is the first demonstration of the Bi-2223 superconducting joint that realized a PC mode LTS/Bi-2223 NMR magnet.

(5) The technological issues have still remained to be addressed. A primary issue is development of a joining procedure applicable for a coil. Another issue is establishment of a mechanical structure and handling method of a Bi-2223 coil with a particular focus on terminal wiring. Addressing these issues will lead to the realization of full-scale PC mode magnets containing Bi-2223 coils, including the 30.5 T NMR magnet.

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