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A new approach to a superconducting joining process for carbon-doped MgB_2 conductor

Dipak Patel¹, Md Shahriar Al Hossain¹, Minoru Maeda²,
Mohammed Shahabuddin³, Ekrem Yanmaz⁴, Subrata Pradhan^{1,5},
Mike Tomsic⁶, Seyong Choi^{7,8} and Jung Ho Kim^{1,8}

¹Institute for Superconducting and Electronic Materials, Australian Institute for Innovative Materials, University of Wollongong, Squires Way, Innovation Campus, North Wollongong, New South Wales 2500, Australia

²Department of Physics, College of Science and Technology, Nihon University, Tokyo 101-8308, Japan

³Department of Physics and Astronomy, College of Science, PO Box-2455, King Saud University, Riyadh - 11451, Saudi Arabia

⁴Department of Mechatronics, Faculty of Engineering and Architecture, Gelisim University, Istanbul-34315, Turkey

⁵Magnet Division, Institute for Plasma Research, Gandhinagar-382428, Gujarat, India

⁶Hyper Tech Research, Inc., 539 Industrial Mile Road, Columbus, Oh 43228, USA

⁷Busan Center, Korea Basic Science Institute, Busan 609-735, Korea

E-mail: jhk@uow.edu.au, sychoi07@kbsi.re.kr and shahriar@uow.edu.au

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Abstract

We report a new approach to a superconducting joining process for unreacted *in situ* carbon (C)-doped magnesium diboride (MgB_2) wires. To operate a magnetic resonance imaging (MRI) magnet in the persistent mode, the superconducting joints between two conductors are as critical as the other key components. In addition, a stable and reliable joining process enables the superconducting magnet to operate without an external power supply. However, joint results using unreacted *in situ* C-doped MgB_2 wires, which are used for high-field operation, have been limited, and only very poor performance has been obtained. By controlling the pressure inside a joint part, in this study, we successfully obtained current carrying retention in the joint of up to 72% compared to wire without a joint. The closed-circuit resistance of our closed-loop coil was less than $1.8 \times 10^{-13} \Omega$ at $16.7 \pm 4.7 \text{ K}$, as measured by the field-decay measurement method. These results indicate that MgB_2 has a promising future in MRI application.

Keywords: C-doped MgB_2 wire, superconducting joints, persistent-mode magnets, MRI application, field-decay measurement

(Some figures may appear in colour only in the online journal)

1. Introduction

Magnesium diboride (MgB_2), which has a critical temperature (T_c) of 39 K, was found to be superconducting in 2001 [1]. Since then, the performance of MgB_2 material and the

manufacturing technology for long-length conductors have been improved significantly [2–7]. Recently, magnet-grade MgB_2 conductors have been commercially available in long-length pieces for employment in various practical applications [5, 6, 8–10]. In particular, the high- T_c of MgB_2 can enable liquid helium (LHe)-free operation up to 25 K. Therefore, there is strong interest worldwide in employing MgB_2 for

⁸ Authors to whom any correspondence should be addressed.

Table 1. Comparison of our joint performance values with various literatures.

Joint details	I_c or current carrying capacity	Joint resistance
Multifilament		
<i>ex situ</i> tape [26]	>300 A at 20 K, self-field	$<10^{-14} \Omega$ at 20 K
<i>ex situ</i> tape (fully reacted) [33]	50 A at 15 K, self-field	$<10^{-9} \Omega$ at 15 K, self-field, four-probe
<i>in situ</i> wire (undoped) [22]	103 A at 10 K, self-field	—
Monofilament		
<i>in situ</i> wire (undoped) [22]	160 A at 20 K, self-field	$<10^{-12} \Omega$ at 10.5 K
<i>in situ</i> wire (undoped) [16]	94 A at 20 K, 2 T	$1.4 \times 10^{-12} \Omega$ at 4.2 K
<i>in situ</i> wire (undoped) [23]	270 A at 20 K, self-field	$<1.3 \times 10^{-10} \Omega$ at 15 K
<i>in situ</i> wire (C-doped) [22]	11 A at 16 K, self-field	—
<i>in situ</i> wire (C-doped) (this work)	>200 A, 20 K, self-field	$<1.8 \times 10^{-13} \Omega$ at 16.7 ± 4.7 K

magnetic resonance imaging (MRI) based on an LHe-free MRI magnet system [11–16]. A special LHe-free MRI system can offer lower principal and maintenance costs, which may make MRI affordable for developing and underdeveloped countries [17].

The ‘open sky MRI’ has already been commercialized using MgB_2 conductors [18]. These MRIs, however, are operated in a driven-mode due to the difficulties in making reliable superconducting joints between MgB_2 conductors. Usually, MRI magnets require a persistent-mode operation to obtain high-quality images [11, 19]. Therefore, for wide applicability of MgB_2 in MRI, more work still needs to be done on the joining process. So far, there have been several reports on joining both *in situ* and *ex situ* MgB_2 conductors [6, 16, 20–27]. Comparison of our joint performance values with various literatures is summarized in table 1.

In the case of *in situ* MgB_2 conductor joints, it has been reported that unreacted monofilament conductor joints can certainly give reliable performance [13, 16, 21–23]. Several coils with a persistent-current switch or joint(s) have been fabricated using an *in situ* monofilament MgB_2 conductor via the wind and react method [13, 16, 21–23, 28]. These coils showed stable persistent-mode operation. It is well known that the high-field performance of *in situ* MgB_2 can be significantly improved by carbon (C)-doping [2, 3, 7, 29–32]. Thus, it is desirable to use C-doped MgB_2 conductor where high in-field performance is required. However, most of the high-performance joint results have been reported for *in situ* MgB_2 conductors without C-dopant for low-field operation (see table 1) [13, 16, 21–23]. Park *et al* reported that the critical current (I_c) of an unreacted *in situ* undoped monofilament wire joint was >230 A at 10 K [22]. On the other hand, when they used an unreacted *in situ* C-doped monofilament wire under the same experimental conditions, the I_c of the joint was only 16 A at 10 K. To the best of our knowledge, there has been no report on high-performance joints using an unreacted *in situ* C-doped monofilament MgB_2 conductor at 20 K. There is thus a need for a superior joining approach for *in situ* C-doped conductors.

In this paper, therefore, a superconducting joining process for unreacted *in situ* C-doped monofilament MgB_2 wires has been investigated in detail, and the persistent-mode

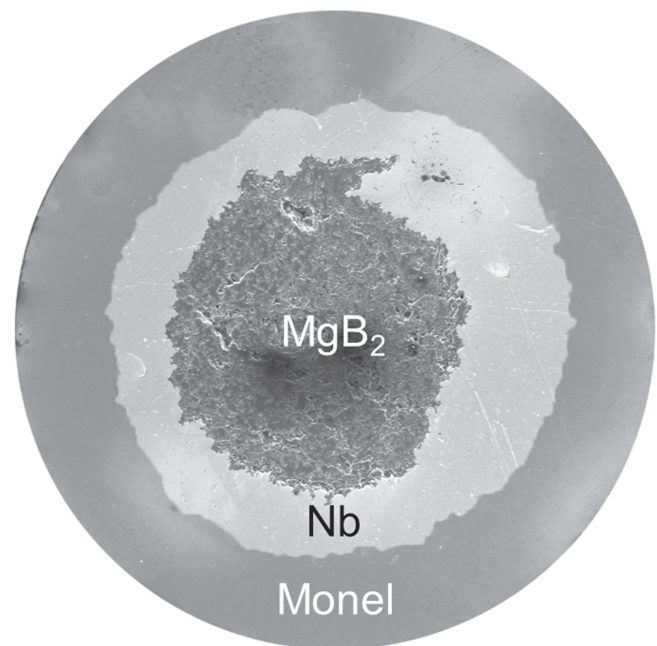


Figure 1. Cross-sectional image of the monofilament MgB_2 wire (Monel sheath, Nb barrier).

performance of the solenoid closed-loop coil was systematically evaluated.

2. Experimental details

A C-doped monofilament MgB_2 wire was fabricated via an *in situ* process by Hyper Tech Research, Inc. [8]. The diameter of the wire was 0.84 mm with a niobium (Nb) barrier and a Monel outer sheath. The superconducting fill factor and the amount of C-dopant in the wire were 21% and 2 wt%, respectively. Figure 1 presents a cross-sectional image of the MgB_2 wire used in this work.

We already reported the joining concept for unreacted *in situ* undoped monofilament MgB_2 wires [16]. More reliable joining using an unreacted *in situ* C-doped MgB_2 conductor is still required, however [22]. In the literature so far, there have been no reports showing superior current carrying capacity (i.e. current carrying retention >60%) with C-doped wire at 20 K. Therefore, it is necessary to study a new approach

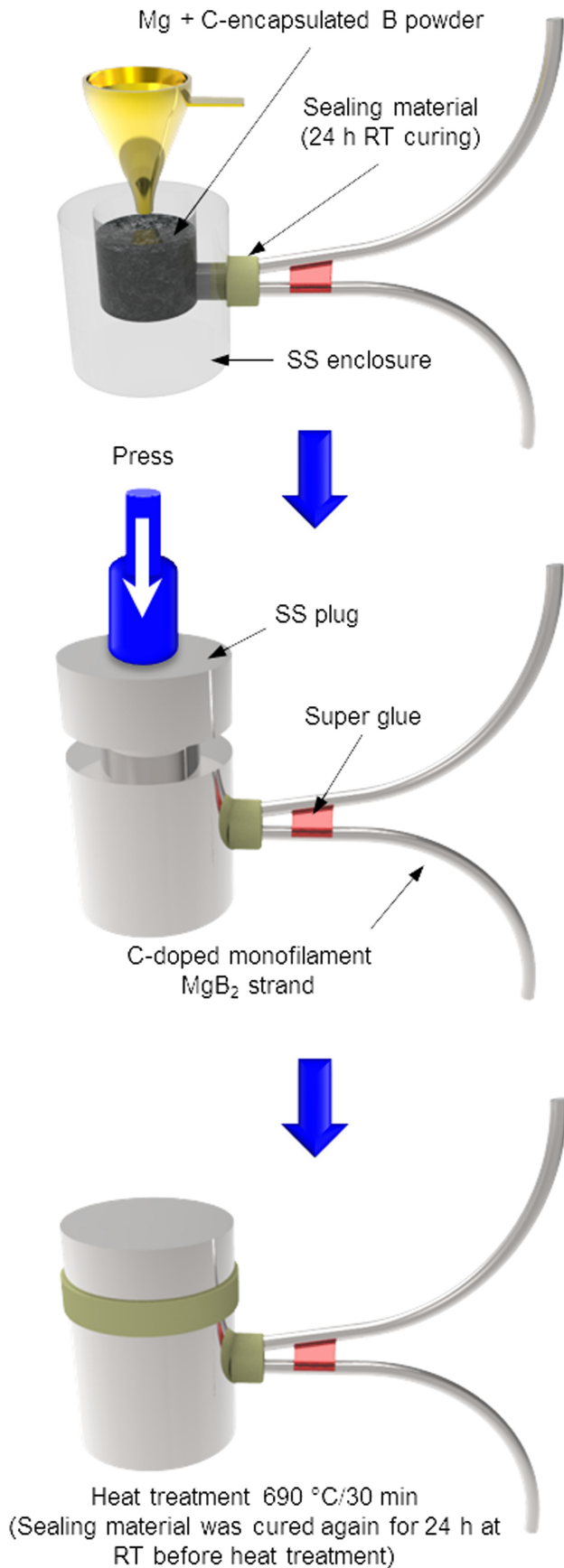


Figure 2. Superconducting joint fabrication process for an unreacted *in situ* C-doped monofilament MgB_2 wire.

to the superconducting joining process for C-doped MgB_2 conductors.

Figure 2 shows the key steps in our modified superconducting joint fabrication process for an unreacted *in situ* C-doped monofilament MgB_2 wire. Firstly, a predefined length (~ 7 mm) of the Monel sheath was chemically etched away using nitric acid from the joining area of the wires. Then, the Nb barrier of the wire (~ 4 mm in length) was partially peeled off until the MgB_2 core was exposed using mechanical polishing. After peeling off the Nb barrier, the exposed MgB_2 cores of two wires were aligned and made to face each other using super glue. As shown in figure 2, the two aligned wires were fixed in a stainless steel (SS) enclosure (inner diameter of 6 mm) using a high-temperature sealing material (Resbond 907GF) [34]. The sealing material was allowed to cure for 24 h at room temperature (RT) in air. In our original joining process, the sealing material needed to be cured at 150°C for 15 min in an oven [16]. For the modified joining process, RT curing of the sealing material can offer better flexibility in handling the joints. After curing the sealing material, previously, packing Mg + 2B powder into the joint was performed in air. In the modified joining process, however, filling in the joint with Mg (99%, 325 mesh, Sigma-Aldrich) + C-encapsulated B (98.8%, ~ 400 nm, C-doping 2 wt%, amorphous, Pavezyum) powder was performed in an argon (Ar) protective atmosphere (in an Ar glove box) to minimize trapping of oxygen while filling the joint with powder [35, 36]. An SS plug was used to compact the powder inside the enclosure (figure 2). The uniaxial pressure for pressing the plug was <0.35 GPa, which was much lower than 0.95 GPa reported in our previous work (i.e., *in situ* undoped monofilament MgB_2 wires) [16]. It should be noted that the applied pressure for the power compaction is more sensitive for C-doped MgB_2 wire joining. In the next step after pressing the powder, the sealing material was applied to the remaining gaps on the SS enclosure, and the joint was then taken out of the Ar glove box. Again, the sealing material was allowed to cure for 24 h at RT (in air) prior to heat-treatment. Finally, the joint was heat-treated in Ar atmosphere at 690°C for 30 min, which are the best conditions for our MgB_2 wire.

The field dependence (up to 2 T) of the I_c of the joint and wire without a joint was investigated using the variable temperature insert (VTI) of an American Magnetics Superconducting (AMS) magnet with direct-current up to 200 A (the maximum power supply capacity) and 20 K operating temperature, using the standard four-probe method with the criterion of $0.1 \mu\text{V cm}^{-1}$ (the criterion of $1 \mu\text{V cm}^{-1}$ is the one that has been most commonly used for MgB_2 conductors).

The joint was then longitudinally cut to take out the MgB_2 powder ingot to perform x-ray diffraction (XRD) analysis. The ingot was ground into a fine powder. The XRD device (Mini-materials Analyser, GBC Scientific Equipment, USA) was used with $\text{Cu K}\alpha$ ($\lambda = 1.54056 \text{ \AA}$) radiation for the XRD analysis [37]. Finally, a solenoid closed-loop coil was fabricated via the wind and react method using the same wire and joining process discussed earlier to evaluate the joint resistance using a field-decay method [16]. The coil with the

Table 2. Specifications of the solenoid closed-loop coil.

Parameters	Specifications
Coil Type	Solenoid (closed-loop)
Wire type	C-doped (<i>in situ</i> mono)
Winding method	Wind and react
No. of joints	1
Inner diameter (mm)	20
Outer diameter (mm)	26.72
Height (mm)	9.24
Turns	42
Computed parameters from FE analysis	
Inductance (μH)	29
Trapped current (A)	104.7
Field at the coil centre (T)	0.22
Pick field on the conductor (T)	0.35
Field on the joint (T)	$3.8 \times 10^{-4} + \text{self-field}$

joint was heat-treated in Ar atmosphere at 690 °C for 30 min. The inductance of the fabricated coil was calculated using the finite element (FE) method implemented with COMSOL Multiphysics software [38]. The specifications of the solenoid closed-loop coil are listed in table 2. After heat-treatment of the coil, the coil was mounted on the test probe. The detailed field-decay measurement set-up is described in our previous paper [16].

The closed-loop coil was inserted into the VTI of the AMS magnet to perform a field-decay measurement. The background magnetic field was increased to 0.24 T with a ramping rate of 10 mT s^{-1} . The closed-loop coil was then cooled down to $\sim 12 \text{ K}$. To induce a current in the closed-loop coil, the background magnetic field was linearly decreased to zero at the ramp-down rate of 0.19 mT s^{-1} . The field-decay in the solenoid closed-loop coil was observed over 48 h.

3. Results and discussion

Figure 3(a) shows the magnetic field dependence of I_c at 20 K for the wire without a joint and for a joint. In self-field at 20 K, the I_c of the joint was $>200 \text{ A}$, so the I_c results are shown from 0.5 T. The self-field performance of the joint at 20 K for the *in situ* C-doped monofilament MgB_2 wire is comparable to the performance of the joint for the *in situ* undoped monofilament MgB_2 wire [22, 23]. The I_c values of the wire without a joint and joint in 0.5 T at 20 K were measured to be 194 A and 140 A respectively. This result means that the performance of the joint was approximately 72% of the performance of our wire without a joint. To the best of our knowledge, such significantly enhanced joint performance at 20 K with *in situ* C-doped MgB_2 monofilament wire is reported in this work for the first time [22]. Nevertheless, the joined wire also showed strong field dependent I_c in the field up to 2 T, and performance fading was also observed. These are preliminary results, so there is still room to further improve the joint performance.

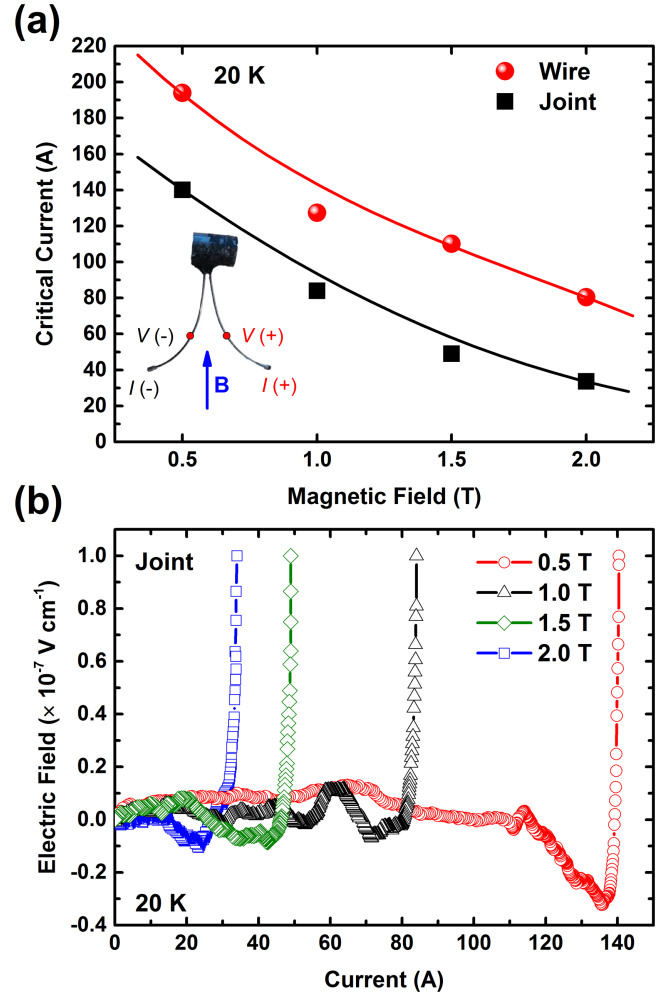


Figure 3. (a) Critical current versus magnetic field characteristics of the wire (i.e. without a joint) and the joint, (b) electric field versus current characteristics of the joint in different magnetic fields at 20 K. The direction of applied magnetic field for I_c measurement is shown in the inset of figure 3(a). The distance between voltage taps on the joint sample was 4 cm (criterion: $0.1 \mu\text{V cm}^{-1}$).

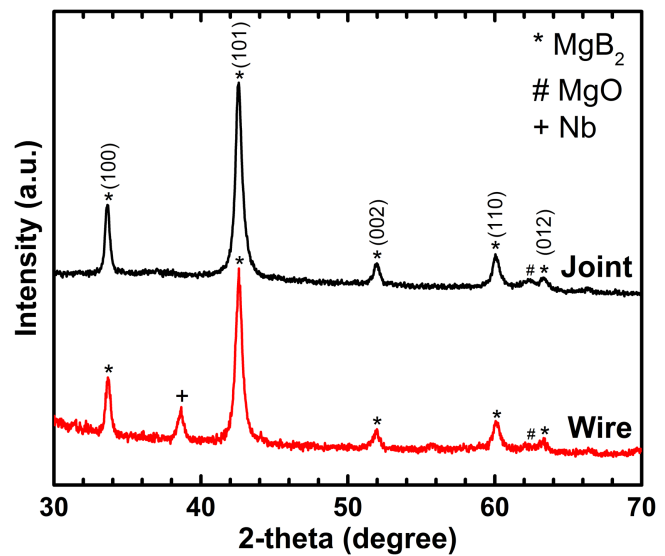


Figure 4. XRD patterns of the MgB_2 powder samples from the wire (i.e. without a joint) and the joint.

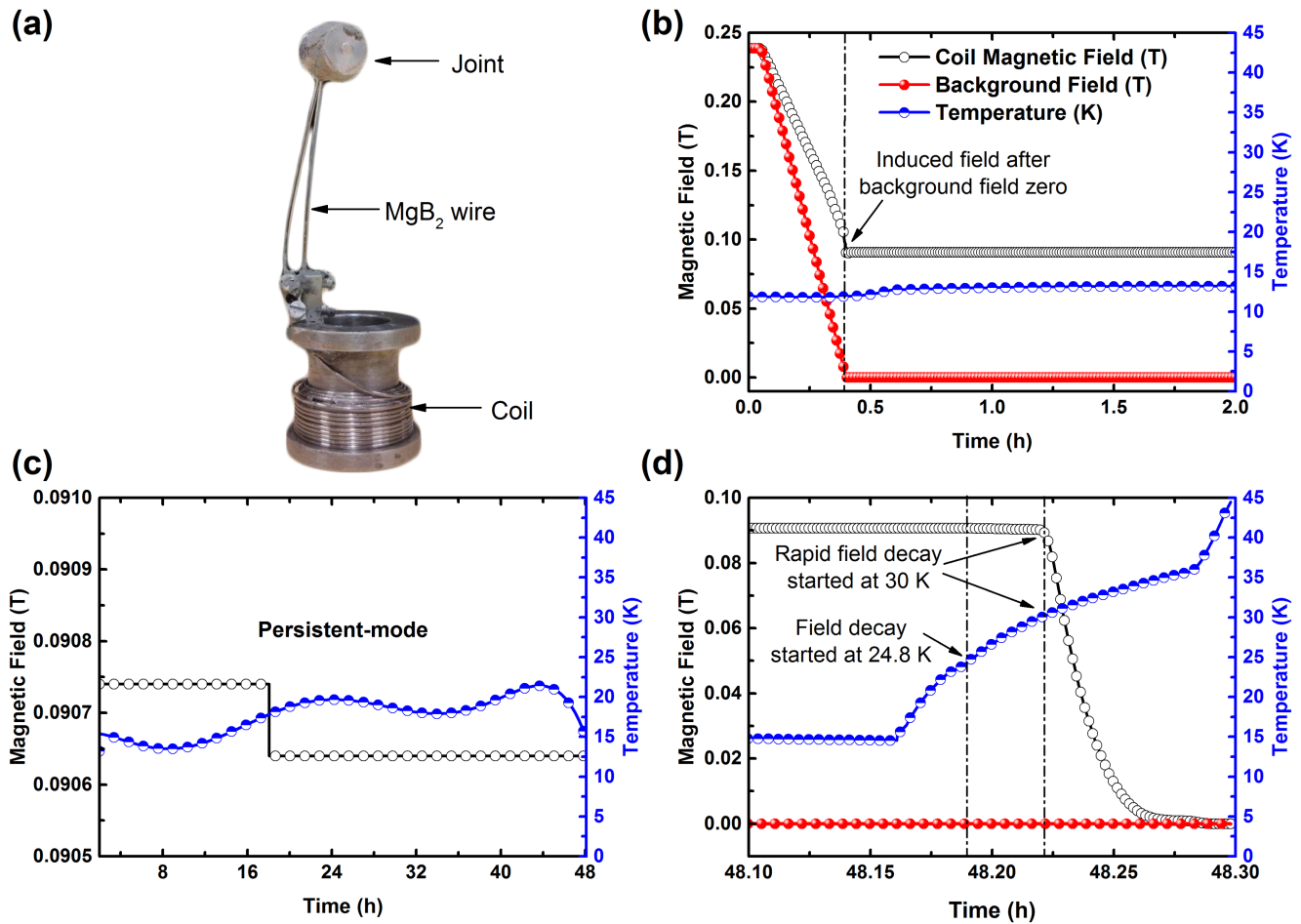


Figure 5. (a) A digital image of the solenoid closed-loop coil, (b) magnetic field (B) and temperature (T) versus time (t) characteristics of the solenoid closed-loop coil during initial induction of the current, (c) B , T versus t characteristics while the coil was in the persistent mode, and (d) B , T versus t characteristics as the temperature of the coil increased after 48 h of persistent-mode operation. The legends in figures 5(b)–(d) are the same.

The electric field versus current characteristics of the joint at 20 K is shown in figure 3(b) as a function of external magnetic field. As can be seen in the figure, the superconducting to normal transition region appears very sharp in the range of 0.01 to 0.1 $\mu\text{V cm}^{-1}$ criterion. This indicates that our joint technique was successful [39].

Furthermore, the joint performance is expected to be strongly dependent on the grain connectivity between the two MgB₂ wires and the phase formation of MgB₂ bulk inside the joint [16]. During the heat-treatment process, the sealing material always remains in close contact with the MgB₂ powder (figure 2) in the joint. Therefore, there was room for the sealing material to diffuse or react with the MgB₂ powder inside the joint during the heat-treatment process. Hence, XRD analysis was carefully carried out to look for impurity phases inside the joined part. Figure 4 shows XRD patterns of powder samples taken out of the wire and the joint. From our results, well developed MgB₂ phase was observed with only a small fraction of MgO. In the XRD pattern of the wire sample, Nb phase was also observed. This Nb might have come from the Nb barrier while we were taking the powder

out from the wire. On the other hand, in the joint powder sample, MgB₂ was a major phase, and no foreign phases existed, except for MgO as a minor phase. The calculated values of MgO fraction in the wire and the joint were 7.7 wt% and 13.3 wt% respectively. These results confirm that the sealing material did not diffuse into or react with the MgB₂ during the heat-treatment process inside the joint.

Under the same experimental conditions, a solenoid closed-loop coil was fabricated to evaluate the precise joint resistance using a field-decay measurement. Figure 5(a) shows a digital image of the solenoid closed-loop coil fabricated in this experiment. Figure 5(b) shows the magnetic field and temperature versus time characteristics of the solenoid closed-loop coil during the initial induction of the current. Before starting the field-decay measurement, the coil temperature was adjusted to ~ 12 K. As can be seen in figure 5(b), when the background field started to decrease, the field began to be induced in the solenoid closed-loop coil. When the background field was decreased to zero, the induced field in the closed-loop coil was 0.09074 T at the Hall probe location (bottom of the coil near the coil winding) [16]. This magnetic

field was equivalent to 104.7 A in the closed-loop coil based on FE analysis. Then, the coil was left in the persistent mode for 48 h (figure 5(c)). Our VTI system has a manual temperature control, so precise temperature control for a very long time period was very difficult to achieve. Hence, during persistent mode, the temperature deviation in the VTI was from 12 K to 21.4 K. Close to 48 h, the temperature of the coil was controlled to ~ 15 K. In the 48 h of persistent-mode operation of the closed-loop coil, 0.1 mT decay in the magnetic field was observed (figure 5(c)). Eventually, at the end of 48 h, as shown in figure 5(d), the temperature of the VTI was increased. The decay of the magnetic field started at 24.8 K, and rapid decay of the magnetic field was observed at 30 K. Some of the computed parameters of the solenoid closed-loop coil during the field-decay measurement are shown in table 2. Therefore, based on 29 μ H inductance and 0.1 mT decay in 48 h, the calculated total circuit resistance of the solenoid closed-loop coil was $<1.8 \times 10^{-13} \Omega$ [16]. This circuit resistance is acceptable for MRI application. These findings show the promising potential of the *in situ* C-doped monofilament MgB₂ wire. Moreover, our technique will pave the way for developing joints for *in situ* and *ex situ* multifilamentary MgB₂ wire/tape conductors.

4. Conclusions

We present a new approach to a superconducting joining process for an unreacted *in situ* C-doped monofilament MgB₂ wire and evaluation of the joint resistance through a field-decay measurement. The new joining approach for C-doped MgB₂ conductor showed significantly enhanced performance compared to reported results to date. According to the literature, our joint fabricated using the unreacted *in situ* C-doped MgB₂ monofilament wire was the first to achieve an I_c value >200 A in self-field at 20 K. Our joint demonstrated current retention of up to 72% in different magnetic fields at 20 K. The results presented here are preliminary results, so there is still room to further improve the joint performance. The XRD analysis showed no diffusion or reaction of the sealing material with the MgB₂ powder inside the joint. The solenoid closed-loop coil showed very reliable persistent-mode operation for a period of 48 h at temperatures up to 21.4 K in self-field. The estimated total circuit resistance was $<1.8 \times 10^{-13} \Omega$ at 16.7 ± 4.7 K temperature. These superconducting joint performance results demonstrate the potential of MgB₂ conductors for MRI application based on an LHe-free system.

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