1-1-2015

## MgB2 superconducting joints for persistent current operation

Dipakkumar Patel
*University of Wollongong*, djp485@uowmail.edu.au

Md S. Hossain
*University of Wollongong*, shahriar@uow.edu.au

Khay Wai W. See
*University of Wollongong*, kwsee@uow.edu.au

Xun Xu
*University of Wollongong*, xun@uow.edu.au

Shaon Barua
*University of Wollongong*, sb201@uowmail.edu.au

*See next page for additional authors*

## Abstract

High-performance superconducting joints are essential for realizing persistent-mode magnets. Herein, we propose a concept and fabrication of such superconducting joints, which yielded reliable performance in the operating temperature range of 4.2-25 K. $MgB_2$-$MgB_2$ joints in magnets are known to result in deterioration of localized electrical, thermal, and mechanical properties. To overcome these problems, the ends of the two wires are inserted into a pellet press, which is then filled with a mixture of unreacted magnesium and boron powders, followed by heat treatment. The critical current capacity and joint resistance were precisely evaluated by the standard four-probe method in open-circuit and by field-decay measurements in a closed-loop, respectively. These joints demonstrated up to 66% of the current-carrying capacity of unjoined wire at 20 K, 2 T and joint resistance of 1.4 x $10^{-12}$ Ω at 4.2 K in self-field.



## Keywords

current, persistent, operation, joints, superconducting, mgb2


## Disciplines

Engineering | Physical Sciences and Mathematics


## Publication Details

Patel, D., Al Hossain, M. Shahriar., See, K. Wai., Xu, X., Barua, S., Ma, Z., Choi, S., Tomsic, M. & Kim, J. (2015). $MgB_2$ superconducting joints for persistent current operation. Superconductor Science and Technology, 28 (6), 065017-1-065017-6.



## Authors

Dipakkumar Patel, Md S. Hossain, Khay Wai W. See, Xun Xu, Shaon Barua, Zongqing Ma, Seyong Choi, Mike Tomsic, and Jung Ho Kim

# $MgB_2$ superconducting joints for persistent current operation

Dipak Patel[1], Md Shahriar Al Hossain[1], Khay Wai See[1], Xun Xu[1], Shaon Barua[1], Zongqing Ma[1], Seyong Choi[2*], Mike Tomsic[3], Jung Ho Kim[1*]

[1] Institute for Superconducting and Electronic Materials, Australian Institute for Innovative Materials, University of Wollongong, Squires Way, Innovation Campus, North Wollongong, New South Wales 2519, Australia

[2] Busan Center, Korea Basic Science Institute, Busan 609-735, Republic of Korea

[3] Hyper Tech Research, Inc., 539 Industrial Mile Road, Columbus, Oh 43228, USA

**E-mail:** jhk@uow.edu.au (J.H. Kim); sychoi07@kbsi.re.kr (S. Choi)

**Abstract**

High-performance superconducting joints are essential for realizing persistent-mode magnets. Herein, we propose a new process and fabrication of such superconducting joints, which yielded reliable performance in the operating temperature range of 4.2 K to 25 K. $MgB_2$-$MgB_2$ joints in magnets, are known to result in deterioration of localized electrical, thermal, and mechanical properties. To overcome these problems, the ends of the two wires are inserted into a pellet press, which is then filled with a mixture of unreacted magnesium and boron powders, followed by heat treatment. The critical current capacity and joint resistance were precisely evaluated by the standard four-probe method in open-circuit and by field-decay measurements in a closed-loop, respectively. These joints demonstrated up to 66% of the current-carrying capacity of unjoined wire at 20 K, 2 T and joint resistance of $< 1.4 \times 10^{-12}$ Ω at 4.2 K in self-field.

## 1. Introduction

Magnetic resonance imaging (MRI) is the key technology for diagnosing critical injuries and diseases. In commercially available MRI systems, superconducting magnets based on niobium titanium (NbTi), are used for producing the high and precise magnetic fields required under persistent-mode for better image quality. It is well known that superconducting MRI magnets are currently operated in expensive liquid helium (LHe) bath at 4.2 K. The soaring LHe prices and possible shortages have increased the demand for LHe-free MRI magnets more than ever [1]. Magnesium diboride ($MgB_2$), which was found to be superconducting in 2001 [2], is considered as a promising candidate for LHe-free operation in MRI due to its relatively low material and fabrication costs compared to high temperature superconductors. In addition, its transition temperature of 39 K allows it to operate at higher temperatures up to 25 K [3-6]. Owing to these benefits [7, 8], there have been many recent reports on $MgB_2$-conductor-based MRI magnets [9-14]. In fact, *PARAmed* has already commercialized LHe-free MRI systems, called as "open sky MRI". These MRIs are not operated in persistent-mode, however. Therefore, a precise power supply is needed, which leads to high-cost operation. In general, MRI magnets are operated in the persistent-mode to retain magnetic field stability throughout the spherical imaging volume, to keep the long-term drift rate of the magnetic field under 0.1 ppm $h^{-1}$, and to maintain overall stable operation [15]. The development of a highly reliable and consistent superconducting joint technique for $MgB_2$ conductors is considered to be the most critical challenge for its wide application in the MRI market.

Several superconducting joint techniques for both *in-situ* and *ex-situ* $MgB_2$ conductors have been reported [13, 16-23]. The first successful joint addressed splicing $MgB_2$ and NbTi for an $MgB_2$ coil operated at 4.2 K under persistent mode, reported in 2005, Hitachi, even if measurement resolution were not high enough to conclude that their joint was indeed superconducting. A real $MgB_2$-$MgB_2$ joint was reported in 2006 by ASG superconductor, based on a field decay measurement of a closed $MgB_2$ loop. Nardelli and co-workers [24] further developed and reported the lowest joint resistance as low as $10^{-14}$ Ω at 20 K for *ex-situ* multifilament $MgB_2$ tape. Most recently in 2013, Ling et al reported their joint concept for *in-situ* monofilament $MgB_2$ wires, resulting in consistent critical current capacities [12], although their closed-loop coil fabricated via the "wind and react" method achieved joint resistance of 1.3 x $10^{-10}$ Ω at 15 K in self-field, which needs to improve by at least an order of magnitude for practical MRI application. It is thus necessary to further improve the state-of-the-art superconducting joint, to give it low joint resistance in an order of $10^{-12}$ Ω for persistent-mode $MgB_2$ magnet operation.

In this study, therefore, of a joint technique for unreacted *in-situ* $MgB_2$ conductor with monofilament, test results at different temperatures and magnetic fields that include comparison of the critical current ($I_c$) of the joint with the wire, the closed-loop coil, and the microstructural analysis of the joints are presented in detail.

## 2. Experimental details

Monofilament $MgB_2$ wire was fabricated by a powder-in-tube (PIT) technique, using an *in situ* process. The detailed method for fabricating wires was reported elsewhere [8]. Magnesium (99 %, 325 mesh) from Sigma Aldrich and amorphous boron (98.8 %, ~ 400 nm) powder from Pavezyum were used as the starting materials with the stoichiometric composition of Mg:B = 1:2. The mixed power was packed into an iron (Fe) tube with an outer diameter (O.D.) of 6.30 mm and an inner diameter (I.D.) of 4.11 mm. The composite wire was swaged and subsequently drawn to an O.D. of 1.00 mm.

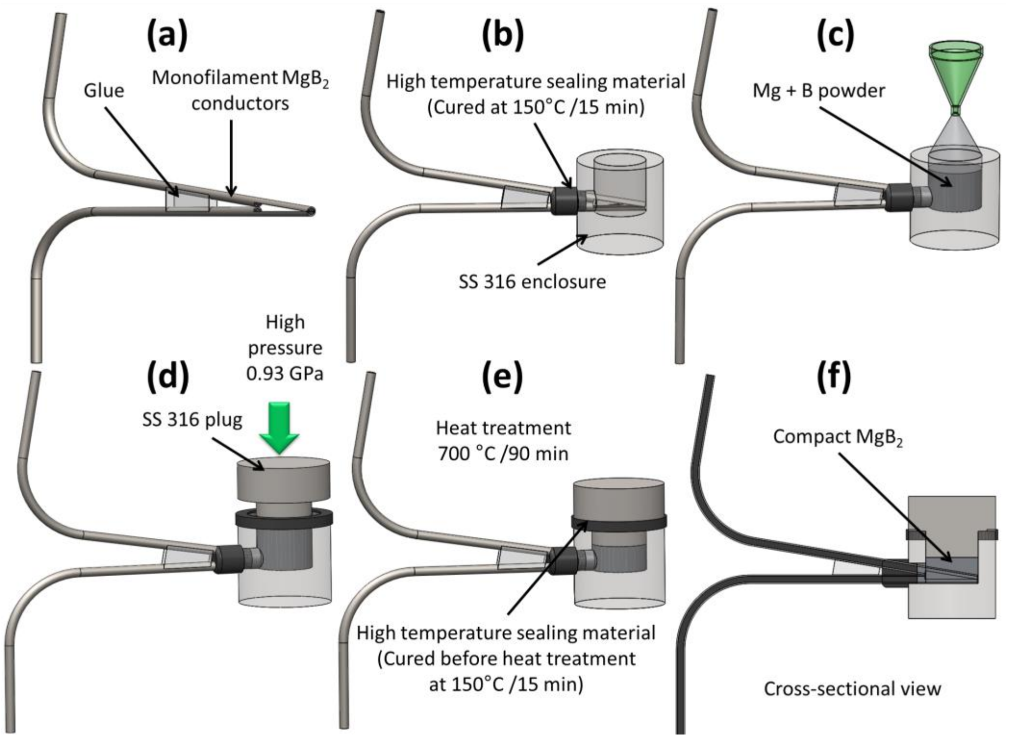


**Figure 1.** Joint configuration and fabrication steps for unreacted monofilament $MgB_2$ wires.

Figure 1 shows the joint configuration and fabrication steps for unreacted monofilament $MgB_2$ wires. As shown in figure 1(a), the metallic sheath material of the two wires was partially peeled off using mechanical polishing until the $MgB_2$ core was exposed. After removing the metallic sheath, exposed cores of the wires were aligned and made to face each other using conventionally available super glue. The two aligned wires were then fixed in a suitable SS316 enclosure having an inner bore diameter of 6 mm, using a high temperature sealing material from Coptaltite, as shown in figure 1(b) [25]. The curing of the sealing material was carried out at 150 °C for 15 min in a drying oven. For the next step, mixed powder from the same batch (Mg + 2B) used for wire fabrication was then packed into the enclosure bore, as shown in figure 1(c). The packed powder density was estimated to be 1.96 g $cm^{-3}$ ± 4 %. As shown in figure 1(d), sealing material was applied on the top edge of the enclosure to hermetically seal the enclosure to avoid Mg evaporation during the heat treatment process. For compacting Mg + 2B powder to make close contact with the wire core, ~ 0.93 GPa of pressure was applied, using a suitable SS316 plug. Again, the curing of the sealing material was carried out at 150 °C for 15 min in the drying oven. Finally, the joints were heat treated in high purity argon (Ar) inert atmosphere at 700 °C for 90 min as shown in figure 1(e). The joints were allowed to cool down naturally to room temperature (RT) before removal from the furnace. Figure 1(f) shows a longitudinal cross-sectional view of the as-prepared joint specimen after a final heat treatment.

The $I_c$s of the joints were measured using an American Magnetics Superconducting (AMS) magnet with DC currents up to 200 A (since 200 A was the limit of the power supply), different temperatures up to 25 K, and magnetic fields in the range of 0 – 2 T, using the standard four-probe method with the criterion of 1 μV $cm^{-1}$.

To observe the longitudinal cross-section, the joints were cut from the appropriate location in the centre of the enclosure. Scanning electron microscopy of all the joints was conducted using a JEOL low-vacuum scanning electron microscope (SEM).

To measure the joint resistance, a small closed-loop coil was fabricated using the same conductor through the "wind and react" method and evaluated using the field-decay method. The selected specifications of the closed-loop coil are listed in table 1. Inductance was calculated using the standard formula because insulation was not used in the closed-loop coil [26]. Later, the two ends of the wire were connected using same joining technique mentioned earlier. The entire coil assembly was heat treated in high purity Ar inert atmosphere at 700 °C for 90 min.

**Table 1.** Selected specifications of the closed-loop coil.

| I.D. (mm) | O.D. (mm) | Height (mm) | Turns | Inductance (L, μH) |
|---|---|---|---|---|
| 20 | 24 | 15 | 28 | 14 ± 7 % |

To determine joint resistance, a field-decay measurement was carried out. The field-decay measurement setup is shown in figure 2. A Hall sensor with 0.1 G sensitivity was installed at the bottom of the closed-loop coil to measure the magnetic field generated by the coil. The cryogenic Cernox$^{TM}$ SD package temperature sensor was also installed using Apiezon® N grease and Kapton® tape for thermally well-coupled with the joint to monitor the temperature [27], during the closed-loop coil measurement as well as short joint measurement (not shown here). The test probe was then inserted into the bore of the variable temperature insert (VTI) of the AMS magnet. First, a magnetization test of the closed-loop coil was carried out to check for any magnetization effect on the coil at RT because Fe was present in the coil as a wire sheath. But, we did not observe any noticeable magnetization effect. External magnet field was applied up to 1.42 T and then decreased to zero with a ramp-down rate of ~ 0.17 T $s^{-1}$. A similar magnetic field and ramp-down rate applied during the actual field-decay measurement to induce current in the closed-loop. Owing to the limitations of our equipment, the field-decay measurement was carried at 4.2 K.

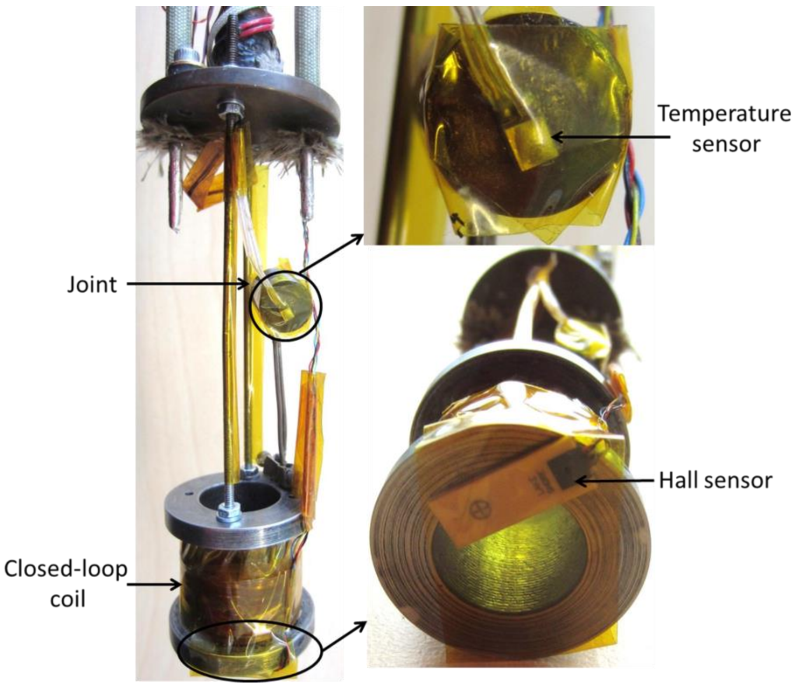


**Figure 2.** Field-decay measurement setup.

## 3. Results and discussion

To evaluate the current-carrying capacity of the jointed $MgB_2$ conductor, firstly, we carried out $I_c$ measurements, as shown in figure 3. Unjoined and jointed wires (inset of figure 3) were heat treated under the same conditions for comparative study. As can be seen in the figure, the $I_c$ values of the unjoined wire under 2 T were measured to be 64 A and 155 A, respectively, at 25 K and 20 K. After mechanical pressing and heat treatment, the jointed wires showed electrical performance deterioration , leading to ~ 35% $I_c$ degradation. The *n*-values, however, which are extracted from the power law of voltage-current characteristic, did not show any noticeable difference between the jointed and unjoined wire. Regardless of the presence or absence of the joint, a very sharp transition appeared near the criterion of 1 μV $cm^{-1}$. Compared to low temperature superconductors such as NbTi and niobium tin ($Nb_3Sn$), however, the *n*-value of the $MgB_2$ conductor still shows a relatively smooth transition from the superconducting state to the resistive one. In particular, it is well-known that both the $I_c$ and the *n*-value are close to the microstructures.

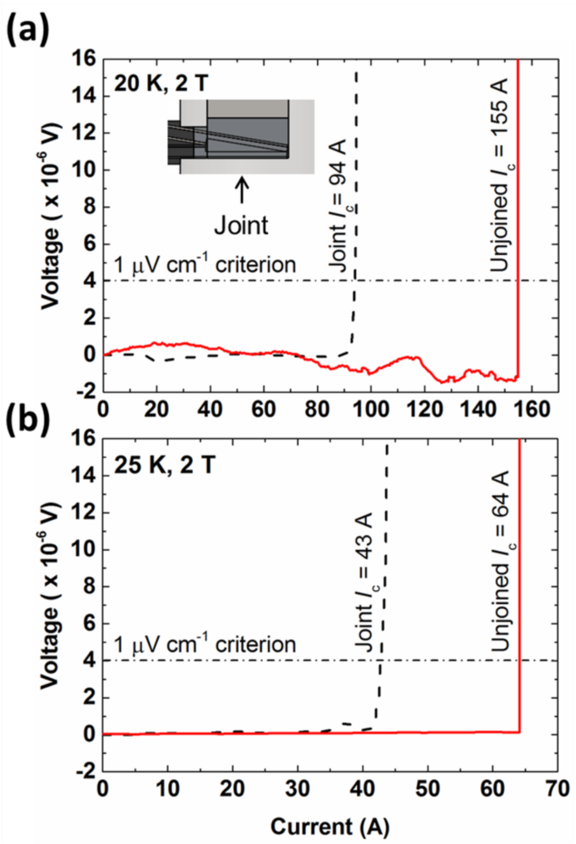


**Figure 3.** Current and voltage characteristics of the jointed (inset figure) and unjoined wires at (a) 20 K, 2 T, and (b) 25 K, 2 T. The distance between voltage taps was 4 cm.

To determine the reason for the current deterioration , a detailed microstructure analysis was conducted using SEM. Figure 4 shows schematic representation of a longitudinal cross-section of a joint and SEM images of the specified areas in a schematic. As has been described, core-exposed $MgB_2$ wires were put into Mg + 2B powders contained in a SS316 enclosure. Thus, we expect that some porous regions might still exist, even after pressing and heat treatment processes. The total length of the wire inside joint was ~ 5.63 mm and direct

$MgB_2$ core to core contact length was ~ 2.38 mm. The remaining length was connected through porous bulk $MgB_2$ between two cores. Figure 4(a) shows schematic representation of a longitudinal cross-section of a joint (not scaled). Figure 4(b) depicts a bulk region formed by the Mg + 2B powders between wire cores. We observed that porous microstructure exists. On the other hand, the interface between wire cores has a denser structure with less porosity, as can be seen in figure 4(c). Most importantly, as can be seen in figure 4(d), the interface between the bulk and the wire core shows different microstructure. This might result in microstructural defects, such as micro-/macro-cracks.

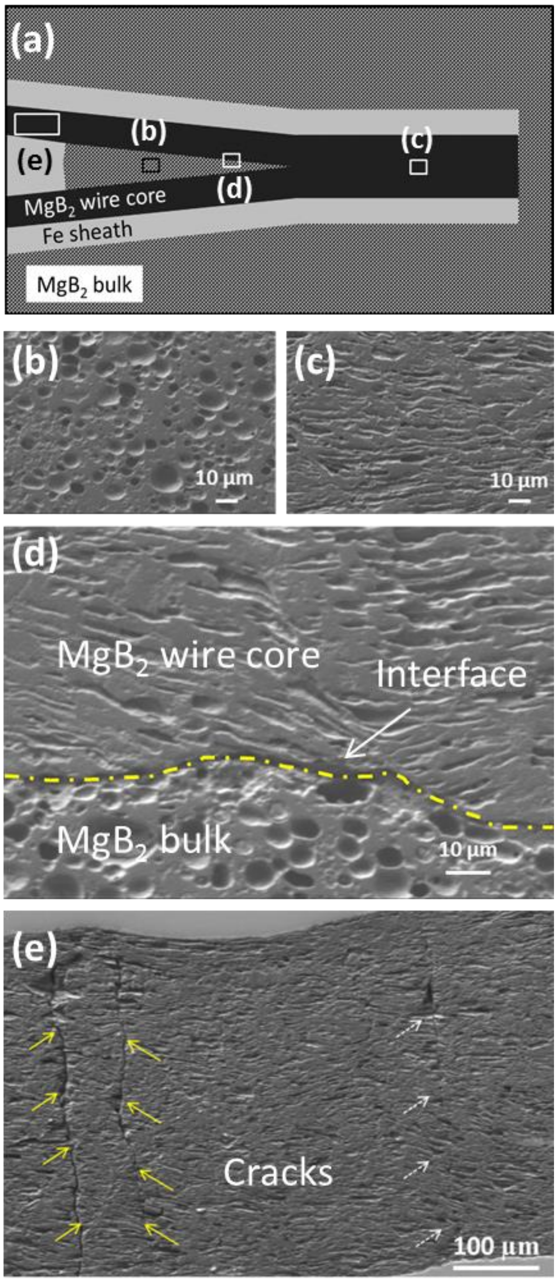


**Figure 4.** (a) Schematic representation of a longitudinal cross-section of a joint (not scaled), SEM images of the jointed wires area represented in a schematic: (b) bulk $MgB_2$ region,

interfaces between (c) $MgB_2$ wire cores, and (d) $MgB_2$ bulk and wire core, and (e) cracks in the joint (yellow and white arrows are showing macro and micro cracks, respectively).

To examine this point, we carried out further microstructure analysis. It is worth noting that pressure was vertically applied to the $MgB_2$ wires. As expected from this, micro-/macro-cracks were observed, as can be seen in figure 4(e). External pressure helps $MgB_2$ formation in the Mg + 2B bulk area, but this can induce microstructural defects, resulting in degradation of current-carrying capacity. In fact, by avoiding the cracks, the performance of the joints can be further improved. During $MgB_2$ formation, micro-/macro-cracks were not healed during the heat treatment process. As a result, we need to determine the optimized pressure conditions.

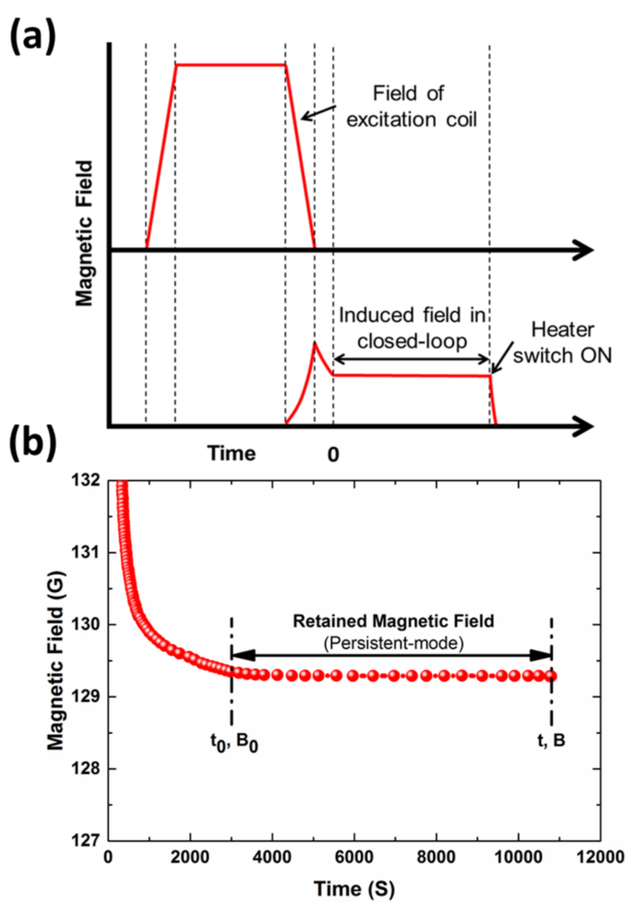


**Figure 5.** (a) The schematic of the operating procedure of the field-decay measurement method. (b) Time decay curve of the captured magnetic field (y-axis field is the induced field in the closed-loop coil).

As was described above, superconducting joint resistance between $MgB_2$ wires can be evaluated by field-decay measurements, i.e., the decay of the induced current in the superconducting closed circuit. The decay behaviour normally has two stages as shown in the schematic of the operating procedure of the field-decay measurement method in figure 5(a). The first stage shows exponentially decreasing current with a high decay rate, due to the lower *n*-value and the small difference between the induced current and $I_c$ of the closed-loop coil [26, 28]. In second stage, the decay is very slow and depends on the joint resistance. The field-decay measurement results obtained from our system are shown in figure 5(b). During entire field-decay measurement period, the joint was exposed to only self-field at 4.2 K. The magnetic field was allowed to stabilize for about 3000 s, and at this point, was considered to be the initial magnetic field $B_0$ at time $t_0$ for the resistance estimation. About 0.1 gauss decay in retained magnetic field was observed in 7764 s. The retained magnetic field after 0.1 gauss decay was 129.3 G which correspond to 30.8 A current based on FEM analysis.

The joint resistance was estimated from temporal decay of magnetic field in the time constant of $L$ - $R$ circuit:

$$B = B_0 \, e^{-\left(\frac{R}{L}\right) t}$$

where $B$ is the magnetic field at time $t$, $B_0$ is the magnetic field at time $t_o$, $L$ is the inductance of the closed-loop coil, $R$ is the joint resistance, and $t$ is the decay time in seconds. The joint resistance was estimated to be $< 1.4 \times 10^{-12}\ \Omega$ at 4.2 K. Based on our experience, the same joint technique will be applied to make an $MgB_2$ based persistent current switch (PCS). For practical MRI application, however, the joint technique without any electromagnetic performance deterioration will be a major challenge for long-term operation.

## 4. Conclusions

We fabricated and evaluated superconducting joints and determined their joint resistance with unreacted $MgB_2$ wire. The $I_c$ results for the jointed wires demonstrated consistent performance, which is promising from the viewpoint of "wind and react" magnets. Despite the performance consistency in the joint performance, about 60 to 66 % of the current was retained after the joint was installed. A field-decay measurement of the closed-loop coil was also conducted to estimate the joint resistance, which is about $< 1.4 \times 10^{-12}\ \Omega$. Optimisation of the wire cutting, heat treatment conditions, and powder density in the joint is required, however, for further performance enhancement. The SEM observations showed very good $MgB_2$ core to core contact in the joint, but some cracks were also induced in this region. These should be avoided for reliable joint processing.

**Acknowledgements**

This work was supported by the Australian Research Council (FT110100170), the KBSI (Korea Basic Science Institute) Grant T35519 and Hyper Tech Research, Inc., USA. The authors are also thankful to Dr. Tania Silver for helpful discussion.

**References**

[1] Wang Z, Van Oort J M and Zou M X 2012 Development of superconducting magnet for high-field MR systems in China *Physica C* **482** 80-6

[2] Nagamatsu J, Nakagawa N, Muranaka T, Zenitani Y and Akimitsu J 2001 Superconductivity at 39 K in magnesium diboride *Nature* **410** 63-4

[3] Canfield P C, Finnemore D K, Bud'ko S L, Ostenson J E, Lapertot G, Cunningham C E and Petrovic C 2001 Superconductivity in dense $MgB_2$ wires *Physical Review Letters* **86** 2423-6

[4] Glowacki B A, Majoros M, Vickers M, Evetts J E, Shi Y and McDougall I 2001 Superconductivity of powder-in-tube $MgB_2$ wires *Supercond Sci Technol* **14** 193-9

[5] Larbalestier D, Gurevich A, Feldmann D M and Polyanski A 2001 High-$T_c$ superconducting materials for electric power applications *Nature* **414** 368-77

[6] Tomsic M, Rindfleisch M, Yue J, McFadden K, Phillips J, Sumption M D, Bhatia M, Bohnenstiehl S and Collings E W 2007 Overview of $MgB_2$ superconductor applications *International Journal of Applied Ceramic Technology* **4** 250-9

[7] Patel D, Hossain M S A, Motaman A, Barua S, Shahabuddin M and Kim J H 2014 Rational design of $MgB_2$ conductors toward practical applications *Cryogenics* **63** 160-5

[8] Patel D, Maeda M, Choi S, Kim S J, Shahabuddin M, Parakandy J M, Hossain M S A and Kim J H 2014 Multiwalled carbon nanotube-derived superior electrical, mechanical and thermal properties in $MgB_2$ wires *Scripta Materialia* **88** 13-6

[9] Li X, Zhang D, Zhang J, Gao Z, Dai S, Zhang Z, Xia D, Zhang G, Wang D, Ma Y, Lin L and Xiao L 2011 A small 1.5 T persistent current operating test magnet using $MgB_2$ wire with high $j_c$ joints *IEEE Trans App Supercond* **21** 1616-9

[10] Modica M, Angius S, Bertora L, Damiani D, Marabotto M, Nardelli D, Perrella M, Razeti M and Tassisto M 2007 Design, construction and tests of $MgB_2$ coils for the development of a cryogen free magnet *IEEE Trans Appl Supercond* **17** 2196-9

[11] Razeti M, Angius S, Bertora L, Damiani D, Marabotto R, Modica M, Nardelli D, Perrella M and Tassisto M 2008 Construction and operation of cryogen free $MgB_2$ magnets for open MRI systems *IEEE Trans Appl Supercond* **18** 882-6

[12] Ling J, Voccio J, Kim Y, Hahn S, Bascunan J, Park D K and Iwasa Y 2013 Monofilament $MgB_2$ wire for a whole-body MRI magnet: Superconducting joints and test coils *IEEE Trans Appl Supercond* **23** 6200304

[13] Park D K, Ling J, Rindfleisch M, Voccio J, Hahn S, Bascunan J, Tomsic M and Iwasa Y 2012 $MgB_2$ for MRI magnets: Test coils and superconducting joints results *IEEE Trans Appl Supercond* **22** 4400305

[14] Yao W, Bascuñán J, Kim W S, Hahn S, Lee H and Iwasa Y 2008 A solid nitrogen cooled $MgB_2$ "demonstration" coil for MRI applications *IEEE Trans Appl Supercond* **18** 912-5

[15] Lvovsky Y, Stautner E W and Zhang T 2013 Novel technologies and configurations of superconducting magnets for MRI *Supercond Sci Technol* **26** 093001

[16] Takahashi M, Tanaka K, Okada M, Kitaguchi H and Kumakura H 2006 Relaxation of trapped high magnetic field in 100 m-long class $MgB_2$ solenoid coil in persistent current mode operation *IEEE Trans Appl Supercond* **16** 1431-4

[17] Penco R and Grasso G 2007 Recent development of $MgB_2$-based large scale applications *IEEE Trans Appl Supercond* **17** 2291-4

[18] Braccini V, Nardelli D, Penco R and Grasso G 2007 Development of ex situ processed $MgB_2$ wires and their applications to magnets *Physica C* **456** 209-17

[19] Li X H, Ye L Y, Jin M J, Du X J, Gao Z S, Zhang Z C, Kong L Q, Yang X L, Xiao L Y and Ma Y W 2008 High critical current joint of $MgB_2$ tapes using Mg and B powder mixture as flux *Supercond Sci Technol* **21** 025017
[20] Yao W, Bascuñán J, Hahn S and Iwasa Y 2009 A superconducting joint technique for $MgB_2$ round wires *IEEE Trans Appl Supercond* **19** 2261-4
[21] Li X, Ye L, Zhang D, Wang D and Ma Y 2010 Joints in $MgB_2$ tapes and wires for persistent current operating magnet *IEEE Trans Appl Supercond* **20** 1528-31
[22] Giunchi G, Saglietti L, Ripamonti G, Albisetti A F, Bassani E and Perini E 2010 Superconducting joints between $MgB_2$ wires and bulks *IEEE Trans Appl Supercond* **20** 1524-7
[23] Wozniak M, Glowacki B A, Setiadinata S B and Thomas A M 2013 Pulsed magnetic field assisted technique for joining $MgB_2$ conductors for persistent mode MRI magnets *IEEE Trans Appl Supercond* **23** 6200104
[24] Nardelli D, Angius S, Capelluto A, Damiani D, Marabotto R, Modica M, Perrella M and Tassisto M 2010 Persistent mode $MgB_2$ short windings *IEEE Trans Appl Supercond* **20** 1998-2001
[25] http://www.copaltite.com
[26] Iwasa Y 2009 *Case studies in superconducting magnets, design and operation issues* (New York: Springer)
[27] http://www.lakeshore.com
[28] Kim J H, Dou S X, Matsumoto A, Choi S, Kiyoshi T and Kumakura H 2010 Correlation between critical current density and n-value in $MgB_2$/Nb/Monel superconductor wires *Physica C* **470** 1207-10