



UNIVERSITY
OF WOLLONGONG
AUSTRALIA

University of Wollongong
Research Online

Faculty of Engineering - Papers (Archive)

Faculty of Engineering and Information Sciences

2007

New Method for the Fabrication of Al-Stabilized Fe/MgB₂ Wires

A. Shcherbakov

University of Wollongong, as695@uow.edu.au

A. V. Pan

University of Wollongong, pan@uow.edu.au

S X. Dou

University of Wollongong, shi@uow.edu.au

E. W. Collings

Ohio State University, USA

<http://ro.uow.edu.au/engpapers/429>

Publication Details

This article was originally published as Shcherbakov, AV, Pan, AV, Dou, SX, and Collings, EW, New Method for the Fabrication of Al-Stabilized Fe/MgB₂ Wires, IEEE Transactions on Applied Superconductivity, 17(2), 2007, 2806-2809. Copyright 2007, IEEE.

Research Online is the open access institutional repository for the University of Wollongong. For further information contact the UOW Library:
research-pubs@uow.edu.au

New Method for the Fabrication of Al-Stabilized Fe/MgB₂ Wires

A. V. Shcherbakov, A. V. Pan, S. X. Dou, and E. W. Collings

Abstract—To employ Al stabilizer on Fe sheathed MgB₂ wire the hot aluminizing technique was used. The present technique allows us to form MgB₂ superconductor by an *in-situ* reaction technique and apply Al stabilizer simultaneously, because the processing temperature of MgB₂ is similar to the melting temperature of Al. Two types of Fe sheathed MgB₂ wires—“reacted” and “green” (unreacted)—were immersed in an Al bath to produce a layer of Al stabilizer on the surface of the superconductor. The magnetic and transport $J_c(B_a)$ curves for both “reacted” and “green” Al/Fe-sheathed wires have been measured at 10 and 20 K and showed the same trend. The concept of Al stabilized Fe/MgB₂ coil fabrication is discussed.

Index Terms—Aluminum stabilized superconductor, fabrication method, hot aluminizing, *in-situ* Fe/MgB₂.

I. INTRODUCTION

SINCE the discovery of superconductivity in MgB₂ [1], this material has been produced in different forms—bulk, tapes and wires. For practical application of superconductors, wires and tapes with high critical current are necessary. Considerable work has been done to investigate and optimize the properties of MgB₂ wires and tapes with different sheath materials such as Ag, Cu, Monel, Ni, Nb, Stainless Steel and Fe (see for example [2]–[10] and references therein). Chemical compatibility with MgB₂, high hardness, low specific weight, and the low price of Fe make it the most suitable sheath material for fabrication of long length MgB₂ wires and tapes. For instance, a critical current density as high as 5×10^5 A/cm² at 20 K and 0 T has been achieved in Fe sheathed MgB₂ wire [11]. However, Fe creates a low thermal conductivity barrier for heat sinking to the cryogen bath or cryocooler. This can result in thermomagnetic instability [12] and trigger the transition from the superconducting to the normal state in superconducting coils at high currents due to avalanche-like heat release.

It is well known that one of the most important goals for practical large current applications of the wires/coils is good thermal stabilization. To minimize thermomagnetic instability in superconducting MgB₂ wires sheathed in iron, a normal

metal with high electrical and thermal conductivity, such as Cu or Al, can be used for stabilization. It has been long recognized that high purity aluminum offers several potential advantages in the stabilization of superconductors. Specifically, aluminum is commercially available with a residual resistance ratio (RRR) in excess of 2500, compared to a maximum of approximately 200 for copper. The magnetoresistance of aluminum also saturates rather quickly, so that the specific resistivity of RRR 2500 aluminum is approximately 1/20 that of RRR 200 copper at 12 T. Aluminum also offers well recognized weight and radiation transparency advantages [13], [14]. Attempts to employ Al stabilizer with LTS superconductors such as NbTi and Nb₃Sn were successful, and these results can be found in the literature (see for example [15]–[18] and references therein). In case of MgB₂, however, direct employment of Al for fabrication of Al-sheathed MgB₂ wire by the *in-situ* process will result in serious damage or even loss of superconductivity in case of a reaction between the Al and the unreacted Mg + 2B powder during the sintering process [20], [21]. On the other hand, the $J_c(B_a)$ performance of Al/MgB₂ *ex-situ* wires will not be high enough, due to the low mechanical strength of the Al (see for example [22] and references therein). Different approaches to the production of Al-stabilized/sheathed MgB₂ wires/tapes with reasonably high $J_c(B_a)$ performance can be found elsewhere [22], [23].

To overcome the problems in the fabrication of Al-stabilized MgB₂ wires that were mentioned above, we have employed the hot aluminizing technique [24]. The present technique allows us to apply Al stabilizer to a completely fabricated and optimized Fe/MgB₂ composite. In this case, Fe plays the role of a barrier preventing diffusion of Al into MgB₂. In addition, the high toughness of Fe enables us to produce a reasonably high-density MgB₂ superconducting core. Furthermore, the hot aluminizing technique can be modified to the simultaneous process of fabrication of Al stabilized wires/coils and *in-situ* MgB₂ formation because the processing temperature of *in-situ* MgB₂ formation (650 °C) is similar to melting temperature of Al (660 °C) [25]. This process is very attractive for the construction of MgB₂ superconducting magnets. In this paper, we report the first results on simultaneous fabrication of *in-situ* Fe/MgB₂ wire and coating this Fe/MgB₂ wire with an Al-layer for stabilization purposes.

II. EXPERIMENTAL PROCEDURE

Fe-sheathed MgB₂ wire has been fabricated using the *in-situ* process and the standard PIT method. An Fe tube with an outer diameter (OD) of 10 mm and a wall thickness of 1 mm was filled with a mixture of pure Mg (99%) and amorphous B (99%) with

Manuscript received August 27, 2006; revised November 28, 2006. This work was supported by the Australian Research Council, Hyper Tech Research Inc., and CMS Alphatech International Ltd.

A. V. Shcherbakov, A. V. Pan, and S. X. Dou are with the Institute for Superconducting and Electronic Materials University of Wollongong, Wollongong, NSW 2522, Australia (e-mail: as695@uow.edu.au).

E. W. Collings is with the Laboratories for Applied Superconductivity and Magnetism Materials Science and Engineering Department, The Ohio State University, Columbus, OH 43210-1179 USA.

Color versions of one or more of the figures in this paper are available online at <http://ieeexplore.ieee.org>.

Digital Object Identifier 10.1109/TASC.2007.899571

the stoichiometry of MgB_2 . The composite was drawn to an OD of 1.4 mm. Several short samples were sintered in a tube furnace at 750°C for 30 min in flowing high purity argon to prevent oxidation. The resultant Fe/MgB_2 with no Al is referred to below as the reference sample. After completing the heat treatment process, the surface of the reacted Fe/MgB_2 wires was plated with Al using the hot aluminizing technique. Short samples of reacted Fe/MgB_2 wire were immersed for 3 min in a molten Al bath kept at a temperature of 750°C under flowing high purity argon. After coating, the sample had an OD of 1.32 ± 0.05 mm. These samples are denoted as the “reacted” Al/Fe/MgB_2 in the text. To investigate the possibility of a simultaneous process for Al plating and *in-situ* MgB_2 formation, short samples of unreacted Fe/MgB_2 wire were immersed for 5 to 30 min in a molten Al bath kept at a temperature of 670 or 770°C under flowing high purity argon. These samples are denoted as the “green” Al/Fe/MgB_2 in the text.

The phase investigation was performed with the help of an x-ray diffractometer with $\text{CuK}\alpha$ radiation. The magnetization loops for the Fe and Al/Fe sheathed MgB_2 wires were measured at 10 and 20 K using a Physical Property Measurement System (PPMS, Quantum Design) with a magnetic field applied perpendicular to the wire long axis. As there is a large sample size effect on the magnetic J_c for MgB_2 fabricated by the reaction *in-situ* process [26], all of the samples for measurement were shaped to the same length of 3.4 mm for comparison. The magnetic J_c was derived from the width of the magnetization loop using Bean’s model. The transport J_c was measured with the four-probe method using a pulsed current source. This technique is described in details in [27]. The voltage contacts were attached directly onto the Al surface with Sn-9%Zn solder.

III. RESULTS AND DISCUSSION

Fig. 1 shows XRD patterns for the core of the “green” Al/Fe/MgB_2 samples after immersing unreacted Fe/MgB_2 wire in an Al bath kept at a temperature of 670°C for 5, 15 and 30 min and at a temperature of 770°C for 5 and 10 min. As can be seen, at lower temperature (670°C) the reaction between Mg and B could be achieved after 30 min. Only a small peak of unreacted magnesium could be found. For the higher temperature Al bath (770°C), the *in-situ* reaction of MgB_2 formation was fully completed in 10 min. A small peak of MgO could be found in all the samples studied. It is to be noted that there is no evidence of any reaction between Al and MgB_2 after the aluminizing routine, indicating the good properties of Fe as a barrier.

SEM investigation of the Al/Fe/MgB_2 wire cross section showed a continuous coating of the Fe sheathed MgB_2 wire with $\sim 10 \mu\text{m}$ thin Al layer (Fig. 2 and Fig. 3). A diffusion layer of Al_3Fe alloy had a thickness of about $9 \mu\text{m}$ at the Al/Fe interface. This diffusion alloy layer indicates good connectivity between the two materials (Fe and Al). It is worth noting that as a result of the hot aluminizing procedure the thickness of the Fe barrier was slightly reduced compared to the Fe/MgB_2 sample due to the reaction between Fe and Al. Increasing the Al bath temperature or time of exposure in hot Al resulted in a further reduction of the Fe barrier thickness (Fig. 3). This is beneficial in case of wire stabilization. As was discussed above, Fe has

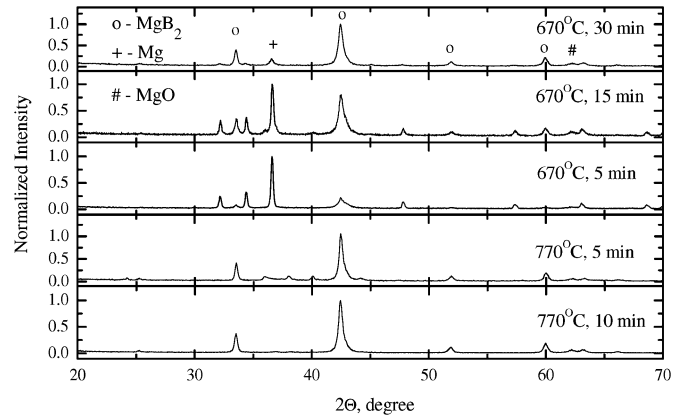


Fig. 1. X-ray diffraction patterns for the “green” MgB_2/Fe wire samples immersed in an Al bath at 670°C for 5, 15 and 30 min and at 770°C for 5 and 10 min.

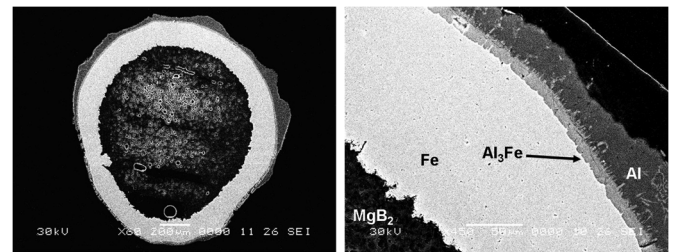


Fig. 2. SEM images of the “reacted” Al/Fe/MgB_2 .

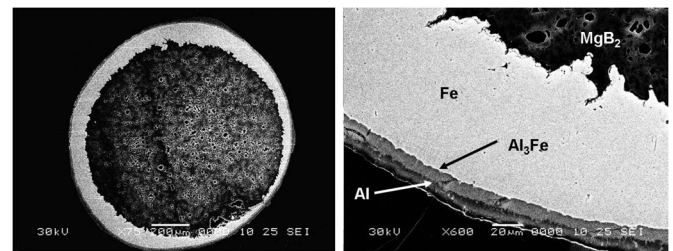


Fig. 3. SEM images of the “green” Fe/MgB_2 wire sample immersed for 10 min in an Al bath kept at 770°C .

undesirably low thermal conductivity. On the other hand, the Fe barrier should prevent the diffusion of Al into the MgB_2 core that may cause severe damage to the superconductor. The formation of the diffusion Al_3Fe alloy may set limits to the desirable Fe barrier thickness and sintering parameters for the hot aluminizing process.

Fig. 4 shows the magnetic J_c values for the reference Fe/MgB_2 , as well as for the “reacted” and “green” Al/Fe/MgB_2 wires with the magnetic field applied perpendicular to the wire long axis. For MgB_2 with the sole Fe-sheath and “reacted” Al/Fe/MgB_2 wire samples magnetic J_c values are similar over the entire range of temperatures and fields measured, although the former is slightly higher than the latter. It is worth noting that the Fe-sheathed MgB_2 wire exhibited more strongly pronounced flux-jump behavior in the low field region (0–2 T) compared to the Al-stabilized MgB_2 wire at 10 K. On the other hand, similar magnetic $J_c(B_a)$ performance indicates that the hot aluminizing process did not affect the properties of the MgB_2 superconducting core. This is a significant advantage of

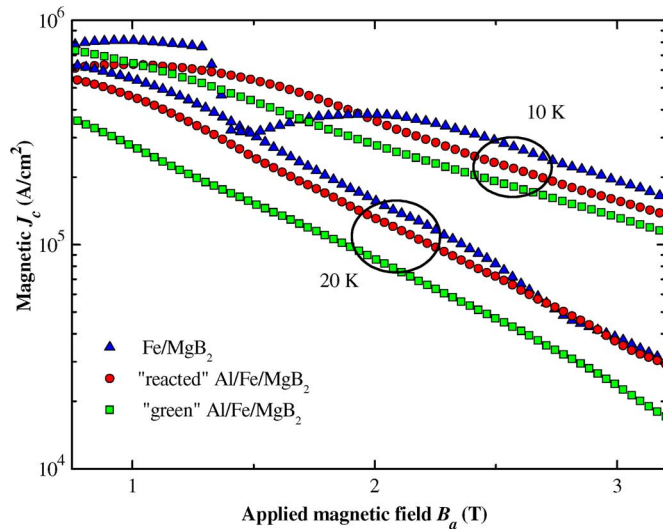


Fig. 4. Magnetic $J_c(B_a)$ curves at 10 K and 20 K for the reference Fe/MgB₂, and for the “reacted” and “green” Al/Fe/MgB₂ wires. The “green” Al/Fe/MgB₂ wire was reacted in a molten Al bath kept at 670 °C for 30 min.

the aluminizing process, which turns out to be a non-destructive approach to thermomagnetic stabilization.

As can be seen from Fig. 4, Al-coating of the Fe-sheathed MgB₂ wires results in better thermomagnetic stabilization at 10 K. Further improvement of stabilization at temperatures below 10 K can be achieved by increasing the Al to superconductor ratio, decreasing the thickness of the Fe barrier, and reduction of the Fe/MgB₂ wire diameter.

It is to be noted that “green” Al/Fe/MgB₂ wire showed a similar trend in the magnetic $J_c(B_a)$ curve to the reference Fe/MgB₂ and the “reacted” Al/Fe/MgB₂ wire, although the $J_c(B_a)$ values are slightly lower (Fig. 4). At this stage, J_c values of 3.6×10^4 and 2.3×10^4 A/cm² have been reached at 20 K and 3 T for the “reacted” and “green” Al/Fe/MgB₂ wires, respectively. These results are in agreement with those for Fe/MgB₂ wires reported by other groups [6], [10].

The transport $J_c(B_a)$ values of the reference Fe/MgB₂, and the “reacted” and “green” Al/Fe/MgB₂ wires are presented in Fig. 5. The $J_c(B_a)$ curves nearly coincide for Fe-sheathed MgB₂ and “reacted” Al/Fe-sheathed MgB₂ wires at $B_a > 4$ T at 10 K, as well as at $B_a > 3.3$ T at 20 K. The transport J_c values of “reacted” Al/Fe/MgB₂ wire are in agreement with previous results on Fe/MgB₂ [5], [7], [28] results, included for comparison in Fig. 5. As can be seen, at 20 K and 3 T the J_c values reached 3.7×10^4 , 3.6×10^4 and 2.2×10^4 A/cm² for sole Fe/MgB₂, and the “reacted” and “green” Al/Fe/MgB₂ wires, respectively.

It is expected that the application of the hot aluminizing technique can be further improved by optimization of the melt-processing temperature and dipping time, Fe sheath thickness, and other parameters. Moreover, the aluminizing technique, which is demonstrated in this work for Fe/MgB₂ wires, can be further extended to fabrication of Al-stabilized Fe/MgB₂ coils. We expect that a coil with a wound Fe-sheathed MgB₂ “green” wire, can be heated up to the MgB₂ reaction temperature. While the reaction is complete or in process, the spool can be immersed in a molten Al bath and cooled down. It is obvious

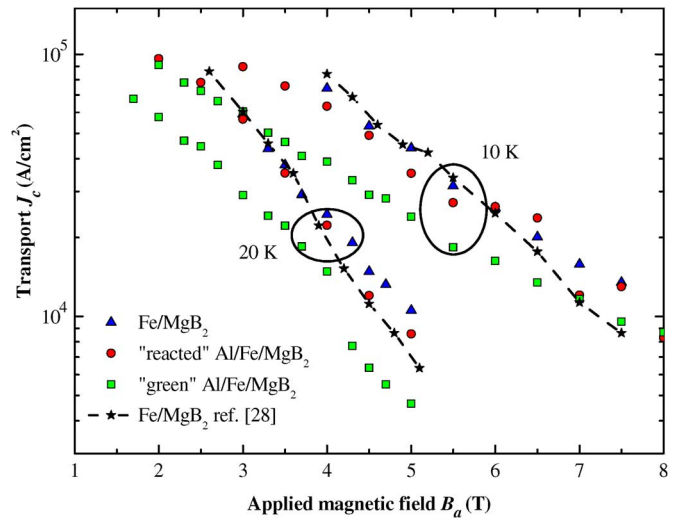


Fig. 5. Transport $J_c(B_a)$ of sole Fe/MgB₂, and the “reacted” and “green” Al/Fe-MgB₂ wires. The “green” Al/Fe/MgB₂ wire was reacted in a molten Al bath kept at 670 °C for 30 min.

that this dual process of *in-situ* MgB₂ formation and coating of Fe/MgB₂ wire/coil by Al will simplify the coil fabrication and markedly improve the cost feasibility of the MgB₂ conductor applications. In addition, Al/Fe/MgB₂ conductors mainly made up of low-density components will be advantageous for airborne, aerospace, and other applications where weight is an important issue.

IV. CONCLUSION

In summary, we have demonstrated a new approach for simultaneous (i) fabrication of *in-situ* Fe/MgB₂ wires and (ii) coating with the Al stabilizer. The transport critical current density of thus-fabricated Al/Fe/MgB₂ wire achieved the level of 2.2×10^4 A/cm² at 20 K and 3.5 T, which is only slightly lower than $J_c = 3.7 \times 10^4$ A/cm² of the reference Fe/MgB₂ sample. The magnetic measurements show that the Al layer stabilizes the Fe/MgB₂ wires in the lower field region at measured temperature range. This method can be easily extended to the production of coils, resulting in reduction of fabrication time and cost of MgB₂ conductor products due to eliminating one processing stage. The light weight of most elements in the Al/Fe/MgB₂ system pave the way for possible use of this superconductor in various applications where weight is a crucial parameter.

ACKNOWLEDGMENT

The authors would like to thank M. Rindfleisch and M. Tomsic for fruitful discussions on this topic, as well as R. Kinnell for help with furnace fabrication.

REFERENCES

- [1] J. Nagamatsu, N. Nakagawa, Y. Zenitani, and J. Akimitsu, “Superconductivity at 39 K in magnesium diboride,” *Nature*, vol. 410, pp. 63–64, 2001.
- [2] S. Jin, H. Mavoori, C. Bower, and R. B. van Dover, “High critical current in iron-clad superconducting MgB₂ wires,” *Nature*, vol. 411, pp. 563–565, May 2001.
- [3] H. Suo, C. Beneduce, M. Dhalle, N. Musolino, J.-Y. Genoud, and R. Flukiger, “Large transport critical currents in dense Fe- and Ni-clad MgB₂ superconducting tapes,” *Appl. Phys. Lett.*, vol. 79, no. 19, pp. 3116–3118, November 2001.

- [4] S. Zhou, A. V. Pan, M. Ionescu, H. K. Liu, and S. X. Dou, "Influence of Ag, Cu and Fe sheaths on MgB_2 superconducting tapes," *Supercond. Sci. Technol.*, vol. 15, pp. 236–240, 2002.
- [5] K. Tanaka, M. Okada, H. Kumakura, H. Kitaguchi, and K. Togano, "Fabrication and transport properties of MgB_2 wire and coil," *Physica C*, vol. 382, pp. 203–206, 2002.
- [6] R. Flukiger, H. L. Suop, N. Musolino, C. Beneduce, P. Toulemonde, and P. Lezza, "Superconducting properties of MgB_2 tapes and Wires," *Physica C*, vol. 385, pp. 286–305, 2003.
- [7] S. X. Dou *et al.*, "Transport critical current density in Fe-sheathed nano-SiC doped MgB_2 wires," *IEEE Trans. Appl. Supercond.*, vol. 13, p. 3199, 2003.
- [8] K. Tanaka, H. Kitaguchi, H. Kumakura, H. Yamada, M. Hirakawa, and M. Okada, "Fabrication and transport properties of an MgB_2 solenoid coil," *Supercond. Sci. Technol.*, vol. 18, pp. 678–681, 2005.
- [9] S. Soltanian, J. Horvat, X. L. Wang, M. Tomsic, and S. X. Dou, "Transport critical current of solenoidal MgB_2/Cu coils fabricated using a wind-reaction in situ technique," *Supercond. Sci. Technol.*, vol. 16, pp. L4–L6, 2003.
- [10] P. Kovac, I. Husek, T. Melisek, M. Kulich, and V. Strbik, " MgB_2 composite wires with Fe, Nb and Ta sheaths," *Supercond. Sci. Technol.*, vol. 19, pp. 600–605, 2006.
- [11] S. X. Dou, A. V. Pan, S. Zhou, M. Ionescu, H. K. Liu, and P. R. Munroe, "Substitution-induced pinning in MgB_2 superconductor doped with SiC nano-particles," *Supercond. Sci. Technol.*, vol. 15, pp. 1587–1591, 2002.
- [12] A. V. Pan and S. X. Dou, "Overcritical state in superconducting round wires sheathed by iron," *J. Appl. Phys.*, vol. 96, no. 2, pp. 1146–1153, July 2004.
- [13] J. M. Royet, J. D. Scudiere, and R. E. Schwall, "Aluminum stabilised multifilamentary NbTi conductor," *IEEE Trans. Magnetics*, vol. MAG-19, no. 3, pp. 761–763, May 1983.
- [14] E. W. Collings, *Applied Superconductivity, Metallurgy and Physics of Titanium Alloys*. New York: Plenum, 1986, pp. 427–433.
- [15] A. Yamamoto, "Advances in superconducting magnets for particle physics," *IEEE Trans. Appl. Supercond.*, vol. 14, no. 2, pp. 477–484, June 2004.
- [16] B. Blau *et al.*, "The CMS conductor," *IEEE Trans. Appl. Supercond.*, vol. 12, no. 1, pp. 345–348, March 2002.
- [17] F. Irie, K. Yamafuji, M. Takeo, and F. Sumiyoshi, "An Al-stabilized Nb_3Sn pulse coil," *IEEE Trans. Magnetics*, vol. MAG-19, no. 3, pp. 672–675, May 1983.
- [18] P. Turowski, L. Z. Lin, and E. Seibt, "Cu- and Al- stabilized Nb_3Sn -multifilamentary conductors and their stability against heat pulses under bath cooling conditions," *IEEE Trans. Magnetics*, vol. MAG-17, no. 5, pp. 2047–2050, September 1981.
- [19] Y. Takahashi *et al.*, "Development of 12 T-10 kA Al-stabilized Nb_3Sn conductor for TMC-II," *IEEE Trans. Magnetics*, vol. MAG-21, no. 2, pp. 157–160, March 1985.
- [20] J. Slusky *et al.*, "Loss of superconductivity with the addition of Al to MgB_2 and a structural transition in $\text{Mg}_{1-x}\text{Al}_x\text{B}_2$," *Nature*, vol. 410, pp. 343–345, March 2001.
- [21] B. Lorenz, R. L. Meng, Y. Y. Xue, and C. W. Chu, "Thermoelectric power and transport properties of $\text{Mg}_{1-x}\text{Al}_x\text{B}_2$," *Phys. Rev. B*, vol. 64, p. 052513, 2001.
- [22] T. Nakane, H. Kitaguchi, and H. Kumakura, "Ex-situ fabrication of MgB_2/Al tapes with high critical current density," *Supercond. Sci. Technol.*, vol. 19, pp. 528–533, 2006.
- [23] S. X. Dou, E. W. Collings, O. Shcherbakova, and A. Shcherbakov, "Aluminium-stabilized magnesium diboride—a new light-weight superconductor," *Supercond. Sci. Technol.*, vol. 19, pp. 333–337, 2006.
- [24] G. A. Moller, US Patent no. 2315725, 1948.
- [25] *2001/2002 CRC Handbook of Chemistry and Physics*, D. R. Lide, Ed., 82nd ed. Boca Raton, FL: CRC Press.
- [26] M. J. Qin, S. Soltanian, X. L. Wang, and S. X. Dou, "On the sample size dependence of the critical current density in MgB_2 superconductor," *Phys. Rev. B*, vol. 69, p. 012507, 2004.
- [27] J. Horvat, S. Soltanian, X. L. Wang, and S. X. Dou, "Magnetic shielding in MgB_2/Fe wires," *IEEE Trans. Appl. Supercond.*, vol. 13, p. 3324, 2003.
- [28] J. H. Kim, unpublished.