RAPID COMMUNICATION

Large field generation with Hot Isostatically Pressed Powder-in-Tube MgB$_2$ coil at 25 K

A. Serquis†‡, L. Civale †, J. Y. Coulter†, D. L. Hammon†, X. Z. Liao†, Y. T. Zhu†, D. E. Peterson†, F. M. Mueller†, V. F. Nesterenko§, and S. S. Indrakanti§

†Superconductivity Technology Center, MS K763, Los Alamos National Laboratory, Los Alamos, NM 87545, USA
§Department of Mechanical and Aerospace Engineering, University of California, San Diego, La Jolla, CA 92093, USA

E-mail: aserquis@cab.cnea.gov.ar

Abstract. We present the fabrication and test results of Hot-Isostatic-Pressed (HIPed) Powder-in-Tube (PIT) MgB$_2$ coils. The coils properties were measured by transport and magnetization at different applied fields ($H$) and temperatures ($T$). The engineering critical current ($J_c$) value is the largest reported in PIT MgB$_2$ wires or tapes. At 25 K our champion 6-layer coil was able to generate a field of 1 T at self-field ($I_c > 220$ A, $J_c \sim 2.8 \times 10^4$ A/cm$^2$). At 4 K this coil generated 1.6 T under an applied field of 1.25 T ($I_c \sim 350$ A, $J_c \sim 4.5 \times 10^4$ A/cm$^2$). These magnetic fields are high enough for a superconducting transformer or magnet applications such as MRI. A SiC doped MgB$_2$ single layer coil shows a promising improvement at high fields and exhibits $J_c > 10^4$ A/cm$^2$ at 7 T.

PACS numbers: 74.70.Ad, 74.60.Jg, 74.62.Bf

‡ on leave from CONICET - Centro Atómico Bariloche, 8400, S.C. de Bariloche, Argentina.
Soon after the discovery of superconductivity in MgB$_2$ [1], it became clear that it has strong potential for commercial applications due to a unique combination of characteristics, such as the high transition temperature $T_c \sim 39$ K, the chemical simplicity, and the low cost of raw materials. In addition, the absence of weak-link behavior at grain boundaries in polycrystalline samples [2] permits the use of simple powder in tube (PIT) methods to fabricate wires [3]. Indeed PIT wires with reasonably good properties were fabricated early on [4, 5, 6, 7], making MgB$_2$ a candidate to replace NbTi or Nb$_3$Sn in magnets. The main applications require high $J_c$ values at high fields and intermediate temperatures, which can be reached by both liquid He as well as commercial cryocoolers. However, the high critical current densities $J_c$ ($\sim 10^6$ A/cm$^2$) usually reported at zero field and $\sim$ 4 K for MgB$_2$ wires or tapes [7] rapidly decrease with increasing temperature or magnetic field. To increase $J_c$, it is necessary to introduce more pinning centers and also overcome the poor connectivity between grains [8]. Besides, the figure of merit of a superconductor not only depends on $J_c$, but also on the engineering critical current density $J_e = f J_c$, where $f$ is the filling factor that reflects the relative ratio of the superconductor to the total cross section of the wire. In this work we present the results of the optimization of several processing parameters to fulfill these requirements not only in short wires but also in the fabrication of coils.

First, an adequate and inexpensive sheath material must be selected. We packed MgB$_2$ powder into stainless steel tubes (inner and outer diameters 4.6 and 6.4 mm) and cold-drew them into round wires with external diameter in the range of 0.8-1.4 mm, with one intermediate annealing. This results in MgB$_2$ cores of very uniform, circular cross section, with diameters of 0.5-0.9 mm corresponding to $f \sim 45\%$ and no reaction between the sheath and the superconductor [8, 9].

A second requirement is to obtain a good inter-grain connectivity by precluding excessive porosity and microcracks, and the formation of large non-superconducting precipitates, such as MgO, at grain boundaries. We have shown [8, 9] that, by adding 5 at.% Mg to the initial powder, heat-treating long lengths of wire with sealed ends to avoid Mg loss, and by choosing appropriate annealing conditions, the microcracks produced by the drawing (the most severe current-limiting factor in the as-drawn wires) can be healed due to a recrystallization promoted by the excess Mg.

Finally, we have found [10, 11] that to improve $J_c$ at high magnetic fields ($H$) and temperatures ($T$), our hot isostatic pressing (HIP) of the PIT wires produces significantly better results than ambient-pressure annealings. In addition to eliminating most of the MgO precipitates, the microcracks, and the porosity, HIPing introduces a high density of crystalline structural defects, including small angle twisting, tilting, and bending boundaries. This results in the formation of subgrains within the MgB$_2$ crystallites with a high dislocation density at subgrain boundaries [11]. These additional pinning centers produce a $J_c$ enhancement (with respect to the ambient-pressure annealings)
Large field generation with Hot Isostatically Pressed Powder-in-Tube MgB$_2$ coil at 25 K

that is marginal at $T = 4$ K and self-field, but becomes very significant as $T$ and $H$ increase, e.g., a factor of $\sim 4$ at $T = 26$ K and $\mu_0 H = 2$ T \[10\]. These short wires achieved the highest reported $H_{irr}$ for PIT MgB$_2$ wires, up to $\sim 17$ T at $T = 4$ K. On the other hand, nano-sized SiC doped MgB$_2$ wires, prepared by Dou et al. \[12\], also show a significant enhancement of critical current density in high magnetic fields over a wide temperature range. Hence, it is interesting to investigate the combined effect of HIPing on SiC doped MgB$_2$ wires.

It is necessary to demonstrate that the high-quality properties mentioned above can be achieved in longer wires. A few groups have reported the fabrication of long wires or tapes for the construction of MgB$_2$ coils or magnets \[13\], but the $J_c$ results are below the values corresponding to short wires. Therefore, we decided to prepare HIPed MgB$_2$ coils capable to produce magnetic fields useful for applications such as MRI, at temperatures compatible with liquid-helium-free operation. Below we report the characteristics and performance of our best coils.

We wound 25 m of our 1 mm diameter, as-drawn PIT wire, around a 3 cm-diameter stainless steel barrel, into a 3.1 cm-long, 4.5 cm-external diameter, 6-layer coil, with insulating fiber-glass fabric intercalated in between layers. The coil was fixed on the outside surface of the barrel to avoid large strain deformation of the wire on the stage of cooling and pressure release due to the difference in coefficients of thermal expansion and elastic moduli of the wire core and the sheath. These strains may result in the fracture of the brittle high density magnesium diboride core. The set was HIPed at 900°C under a pressure of 200 MPa for 30 min, depressurized, and cooled at 5°C/min to room $T$. For measurements, we removed the coil from the barrel, made $\sim 15$ cm long current contacts by soldering a Cu tape, added two voltage contacts (placed about 24 m apart on the wire) to measure the I-V curves, attached a Hall probe at the coil center, and inserted the coil coaxially in the 5 cm bore of a 9 T superconducting magnet.

We tested the coil both immersed in liquid He (4 K) and in liquid Ne (24.6 K $< T < 26.5$ K). Fig. 1 shows (in solid symbols) the maximum current that we were able to put through the coil as a function of applied field ($H^a$). At 4 K a break in the $I_c(H^a)$ curve is visible at $\sim 7$ T, which coincides with a clear change in the I-V curves. Above 7 T the I-V curves were smooth and had a well behaved n-value, indicating that we were correctly measuring $I_c$, while at lower $H^a$ the voltage suddenly jumped from almost zero to a large value, implying a quench of the coil due to the heat propagating from the current contacts. At 25 K (pumped liquid Ne) we were always in that situation (regardless of this, for simplicity we call the maximum current $I_c$ in all cases). The solid and dotted lines are the $I_c$ obtained from magnetization measurements using the Bean model on a 0.5 cm long piece of the wire. The excellent coincidence of the magnetization and transport $I_c$ at 4 K above 7 T indicates that the 25 m long wire is very homogeneous. Indeed, the performance is as good as that of our short HIPed wires
Large field generation with Hot Isostatically Pressed Powder-in-Tube MgB$_2$ coil at 25 K

previously reported \cite{10}.

For each ($H^a, T$) in Fig. 1, we used the Hall probe to measure the total field at the coil center when a current $I_c$ was applied (open symbols). The field difference between the open and solid symbols at a given $I_c$ and $T$ (connected by arrows in the figure) is the field generated by the coil ($H^{gen}$). We found excellent proportionality $H^{gen} = (4.5 \text{ mT/A}) \times I_c$ (dashed line in fig. 1). At 4 K we were limited by the maximum current of the power supply, 350 A, which generated a field $H^{gen} = 1.6 \text{ T}$ at $H^a = 1.25 \text{ T}$, resulting in a total field at the coil center of 2.85 T and corresponds to a $J_c \sim 4.5 \times 10^4 \text{ A/cm}^2$. The intersection of the solid and dashed lines indicate that at 4 K and $H^a = 0$ this coil could generate a field of $\sim 2.5 \text{ T}$, at a current of $\sim 550 \text{ A}$.

We also measured $I_c(T)$ and $H^{gen}(T)$ (with the Hall probe) at $H^a = 0$, by pumping the liquid Ne, as shown in Fig. 2. At $T = 26.5 \text{ K}$ (ambient pressure at Los Alamos), $H^{gen} = 0.85 \text{ T}$, and at $T = 24.6 \text{ K}$ (slightly above the Ne triple point) $H^{gen}(24.6 \text{ K}) = 1 \text{ T}$. This correspond to a $J_c \sim 2.8 \times 10^4 \text{ A/cm}^2$.

Another single layer coil was made following the same procedure with a 4 meter length wire that contains MgB$_2$ + 5 % SiC, to explore the effect of microprecipitates in combination with the HIPing process. Fig. 3 displays (in solid circles) the maximum current that we were able to put through the coil as a function of applied field together with the data of the 6-layer coil (in solid squares). For easy comparison with other published values, the right axis shows $J_c$ as a function of field (both coils were built with wires of the same diameters). The main result is that the critical current densities measured by transport are at least twice larger than those of the best samples without SiC (i.e: $J_c \sim 1 \times 10^4 \text{ A/cm}^2$ at 7 T). Again due to heating problems at the contacts we could get reliable data only for applied magnetic fields above $H=6.5 \text{ T}$. The solid and dotted lines are the $I_c$ obtained from magnetization measurements using the Bean model on 0.5 cm long pieces of the doped and un-doped wires, respectively.

In summary, we built a 6-layer, 4.5 cm-external diameter, 3.1 cm-long coil, by winding 25 m of 1 mm-diameter powder-in-tube MgB$_2$ wire and subsequently hot isostatic pressing. At $T = 4 \text{ K}$ and $H^a = 1.25 \text{ T}$ it generates a field of 1.6 T (total field at the coil center 2.85 T). At $T = 24.6 \text{ K}$ and $H^a = 0$ the generated field was $H^{gen}(24.6 \text{ K}) = 1 \text{ T}$. The performance of this compact coil satisfies the requirements for use in liquid-helium-free MRI systems. The developed method can be scaled to process coils with diameter about 1 meter. We also explored the combination of HIPing with SiC doping and we found a significant improvement of $J_c$ for high fields.
Large field generation with Hot Isostatically Pressed Powder-in-Tube MgB$_2$ coil at 25 K

References

[1] Nagamatsu J, Nakagawa N, Muranaka T, Zenitani Y and Akimitsu J 2001 *Nature (London)* **410** 63
[2] Bugovslavsky Y, Perkins G K, Qi X, Cohen L F and Caplin A D 2001 *Nature (London)* **411** 561
[3] Jin S, Mavoori H, Bower C and van Dover R B 2001 *Nature (London)* **411** 563
[4] Suo H L, Beneduce C, Dhallé M, Musolino N, Genoud J Y and Flükiger R 2001 *Appl. Phys. Lett.* **79** 3116
[5] Fujii H, Kumakura H and Togano K 2002 *J. Mater. Res.* **17** 2339
[6] Soltanian S, Wang X L, Kusevic I, Babic E, Li A H, Qin M J, Horvat J, Liu H K, Collings E W, Lee E, Sumption M D and Dou S X 2001 *Physica C* **361** 84
[7] Flükiger R, Suo H L, Musolino N, Beneduce C, Toulemonde P and Lezza P 2003 *Physica C* **385** 286 and references therein
[8] Serquis A, Civale L, Hammon D L, Coulter J Y, Liao X Z, Zhu Y T, Peterson D E and Mueller F M et al. 2003 *Appl. Phys. Lett.* **82** 1754
[9] Serquis A, Civale L, Hammon D L, Liao X Z, Coulter J Y, Zhu Y T, Peterson D E and Mueller F M 2003 *J. Appl. Phys.* **94** 4024
[10] Serquis A, Civale L, Hammon D L, Liao X Z, Coulter J Y, Zhu Y T, Jaime M, Peterson D E, Mueller F M, Nesterenko V F and Gu Y 2003 *Appl. Phys. Lett.* **82** 2847
[11] Liao X Z, Serquis A, Zhu Y T, Civale L, Hammon D L, Peterson D E, Mueller F M, Nesterenko V F and Gu Y et al. 2003 *Supercond. Sci. Technol.* **16** 799
[12] Dou S X, Soltanian S, Horvat J, Wang X L, Munroe P, Zhou S H, Ionescu M, Liu H K and Tomsic M 2002 *Appl. Phys. Lett.* **81** 3419; Zhou S H, Pan A V, Qin M J, Liu H K and Dou S X 2003 *Physica C* **387** 321
[13] Grasso G, Malagoli A, Modica M, Tumino A, Ferdeghini C, Siri A S, Vignola C, Martini L, Previtali V and Volpini G 2003 *Supercond. Sci. Technol.* **16** 271; Machi T Shimura S, Koshizuka N and Murakami M 2003 *Physica C* **392**, Pt. 2 1039; Tanaka K, Okada M, Kumakura H, Kitaguchi H and Togano K 2002 *Physica C* **382** 203
Large field generation with Hot Isostatically Pressed Powder-in-Tube MgB$_2$ coil at 25 K

Figure 1. Critical current $I_c$ (left axis) and engineering critical current density $J_e$ (right axis) vs. magnetic field at $T = 4$ K and 25 K of the 6-layer coil. The full symbols indicate the external field applied by the 9T magnet. The open symbols indicate the total field at the coil center. The difference (indicated by arrows) is the field $H^{\text{gen}}$ generated by the coil. Solid and dotted lines: magnetization data for a short piece of the coil wire. Dashed line: $I_c$ vs. $H^{\text{gen}}$ relation.
Figure 2. Critical current (left axis) and field generated by the coil (right axis) vs. temperature of the 6-layer coil.
Figure 3. Critical current $I_c$ (left axis) and critical current density $J_c$ (right axis) vs. magnetic field at $T = 4$ K for the 1-layer SiC doped MgB$_2$ coil. The inset shows a photograph of the 1-layer coil. For comparison we also included the 6-layer coil data. Solid and dotted lines: magnetization data for short pieces of the doped and un-doped wires, respectively.