

High upper critical field and irreversibility field in MgB₂ coated-conductor fibers

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We report on structural and superconducting properties of round MgB₂ coated-conductor fibers deposited by hybrid physical-chemical vapor deposition on SiC fibers. The coating is polycrystalline and composed of elongated crystallites with dimensions less than 1 μm in length and 0.2 μm in width. The pure MgB₂ fiber shows a zero-resistance T_c of 39.3 K. The carbon-alloyed fibers show a high upper critical field of 55 T at 1.5 K and a high irreversibility field of 40 T at 1.5 K. The result demonstrates great potential of MgB₂ coated conductors for superconducting magnets. © 2005 American Institute of Physics. [DOI: 10.1063/1.2149289]

The recently discovered superconductor MgB₂ (see Ref. 1) is a promising material for high-magnetic-field applications.² The transition temperature at 40 K allows practical operation above 20 K using cryocoolers. Unlike high temperature superconductors where critical current density J_c drops sharply across the grain boundary when the grains are misaligned, grain boundaries do not degrade J_c in MgB₂.^{3,4} Wires and tapes of MgB₂ have been made using the so-called “powder-in-tube” (PIT) technique with encouraging results.^{5–7} MgB₂ conductors also promise lower cost.³ Recently, we have shown record high values of upper critical field $H_{c2}(0)$ over 60 T in textured carbon-alloyed MgB₂ thin films produced by hybrid physical-chemical vapor deposition (HPCVD).⁸ Whether such record high values can be achieved in forms more suitable for practical conductors is of great interest. Here we report on polycrystalline carbon-alloyed MgB₂ coated-conductor fibers with high H_{c2} (the field at which a superconductor becomes normal) and high irreversibility field H_{irr} (the field at which a superconductor ceases to carry supercurrent). The result demonstrates great potential of the coated conductor approach towards MgB₂ high-field wires for superconducting magnets.

Coated conductors have been widely studied for high temperature superconductors.^{3,4} Coated conductors have also been explored for MgB₂ by sputtering on Hastelloy⁹ and by electroplating on stainless steel.¹⁰ These works have generated H_{c2} and H_{irr} values similar to those of PIT MgB₂ wires. In this work, the MgB₂ coated conductors were grown by HPCVD¹¹ on round SiC fibers with a tungsten core.^{12,13} De-

tails of the HPCVD process have been described elsewhere.¹¹ The SiC/W fibers, about 1 cm in length, were placed on the top horizontal surface of the susceptor (heater) and heated to 685–720 °C in H₂ carrier gas during the deposition. Heated Mg chips placed near the SiC fibers served as the Mg source while B₂H₆ was the B source. The deposition rate was about 0.1 μm/min at 700 °C. Some fibers were coated multiple times to achieve thicker coating. For the deposition of the carbon-alloyed MgB₂ coating, we added bis(methylcyclopentadienyl)magnesium [(MeCp)₂Mg], a carbon-containing metalorganic magnesium precursor, to the gas flow during the deposition, and the carbon content in the coating was controlled by the flow rate of a secondary hydrogen flow passing through the (MeCp)₂Mg bubbler.¹⁴

Figure 1 shows scanning electron microscope (SEM) images of a round MgB₂ coated-conductor fiber. The diameter of the SiC fiber is ~100 μm with a tungsten core of ~10 μm.¹³ Except for a defect spot, Fig. 1(a) shows a uniform coating of the fiber. Figure 1(b) shows that the SiC fiber has a columnar structure along the radial direction.¹² The MgB₂ coating, seen as the bright layer on the surface of the fiber, is about 0.75 μm in thickness. Although the fiber was coated only once, it shows a relatively uniform thickness of the MgB₂ layer without shadow effect, which is due to the high gas pressure (100 Torr) and the diffusive nature of the deposition process. Figure 1(c) is a blown-up view that shows more clearly the microstructure of the coating. The MgB₂ coating has a granular structure with randomly oriented crystallites. The grains are elongated with length less than 1 μm and width of 0.2 μm. This microstructure is very similar to those in some early polycrystalline MgB₂ films.¹⁵

The structure of the MgB₂ coating was further studied by x-ray diffraction using a two-dimensional area detector. The integrated area diffraction θ – 2θ scan is shown in Fig. 2. The peaks from the SiC fiber, marked by “*,” indicate that SiC is

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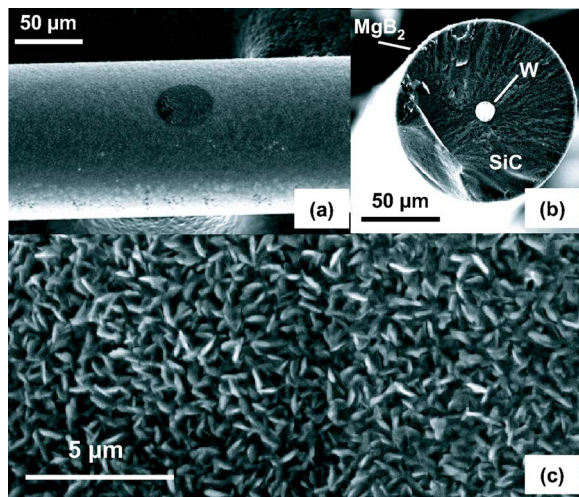


FIG. 1. (a) Scanning electron microscope (SEM) images of a MgB_2 coated-conductor fiber. (b) A cross-section image of the MgB_2 coated-conductor fiber. (c) A blown-up SEM image of the surface, showing elongated MgB_2 crystallites with random orientations.

of the 3C polytype (β -SiC, zinc blende structure).¹⁶ Several diffraction peaks off different MgB_2 planes are seen, indicating that the MgB_2 crystallites are oriented randomly. The polycrystalline nature of the microstructure and the small grain size make the diffraction intensity from MgB_2 weak. The lattice constants of the MgB_2 layer are $a = 3.078 \pm 0.01 \text{ \AA}$ and $c = 3.514 \pm 0.005 \text{ \AA}$, matching very well with the bulk values of pure MgB_2 .¹ Also observed are diffraction peaks due to Mg_2Si . It could be the result of reaction of Mg with SiC¹⁷ or with the free Si existing in the SiC fiber.¹³

The superconducting properties of the coated-conductor fibers are similar to those in epitaxial pure MgB_2 and textured carbon-alloyed MgB_2 films deposited by HPCVD. For example, a pure MgB_2 fiber shows a zero-resistance transition temperature T_c of 39.3 K. It has a residual resistivity of $\rho_0 \sim 30 \mu\Omega \text{ cm}$, much higher than that in the clean epitaxial HPCVD MgB_2 films ($\rho_0 \sim 0.28 \mu\Omega \text{ cm}^{18}$), and a residual resistance ratio of $RRR = R(300 \text{ K})/R(50 \text{ K}) = 2.54$, much smaller than that in the clean films ($RRR \sim 30^{18}$). This is due to the granular nature of the layer and the existence of semi-conducting Mg_2Si , presumably at the grain boundaries.

Measurements in magnetic field were carried out at the National High Magnetic Field Laboratory at Florida State

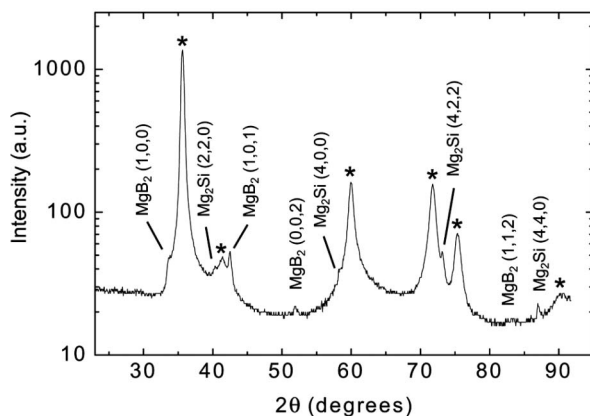


FIG. 2. Integrated x-ray diffraction θ - 2θ scan of a pure MgB_2 coated-conductor fiber collected using a two-dimensional area detector.

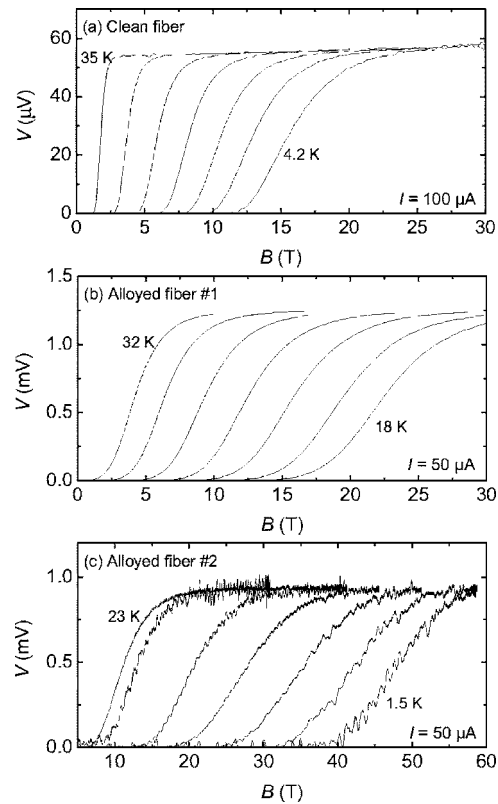


FIG. 3. Voltage-magnetic field curves for (a) a pure MgB_2 fiber, (b) carbon-alloyed MgB_2 fibers 1, and (c) carbon-alloyed MgB_2 fibers 2. For each set of data, the lowest and the highest temperatures of the measurements are shown.

University (dc field, up to 30 T) and Los Alamos National Laboratory (pulsed field, up to 60 T). Figure 3 shows voltage versus applied magnetic field curves measured at different temperatures for three MgB_2 coated-conductor fibers: (a) a pure clean MgB_2 fiber, (b) carbon-alloyed MgB_2 fiber 1, and (c) carbon-alloyed MgB_2 fiber 2. The measurement currents are 100, 50, and 50 μA for the three fibers, respectively. Carbon alloying suppresses T_c and increases resistivity of the coating, leading to larger voltage signals in the carbon-alloyed MgB_2 fibers than in the pure MgB_2 fiber. From these curves, H_{c2} and H_{irr} are determined by the following criteria: $R(H_{c2}) = 0.9R_0$ and $R(H_{irr}) = 0.1R_0$, where R_0 is the residual resistance above the superconducting transition.

Previously, we have shown textured films of carbon-alloyed MgB_2 prepared by HPCVD H_{c2} of 51 T in parallel field and 35 T in perpendicular field at 4.2 K.⁸ Since the MgB_2 grains in the coated-conductor layers are randomly oriented, MgB_2 grains with a - b grains parallel to the applied field become normal at a higher field than those whose a - b grains are perpendicular to the applied field, with grains of other orientations in between. Because H_{c2} measures the onset of superconductivity, the measured H_{c2} values in the coated-conductor fibers should be similar to the higher, parallel-field H_{c2} in the textured films. On the other hand, as soon as the superconducting MgB_2 grains form a continuous path, the fiber will reach zero resistance, which is measured by the H_{irr} values in the coated-conductor fibers.

Figure 4 shows the temperature dependencies of (a) H_{c2} and (b) H_{irr} for the three fibers in Fig. 3. The clean fiber has an H_{c2} (4.2 K) of 20 T, which is dramatically enhanced by carbon alloying. At $T = 1.5 \text{ K}$, alloyed fiber 2 shows an H_{c2} of

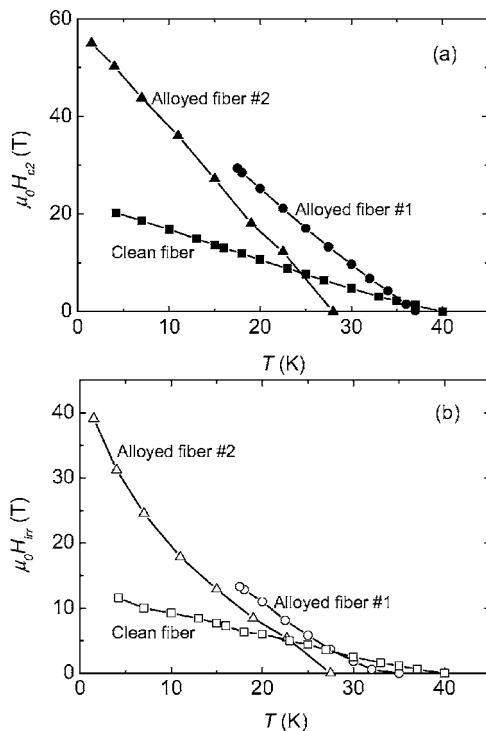


FIG. 4. Temperature dependencies of (a) H_{c2} and (b) H_{irr} for the three MgB_2 coated-conductor fibers shown in Fig. 3.

55 T, and at $T=25$ K, alloyed fiber 1 has an H_{c2} of 17 T. The enhancement of H_{c2} by carbon alloying can be qualitatively explained by the modifications of intraband and interband scattering in the two (σ and π) bands of MgB_2 .¹⁹ Similar dramatic enhancement by carbon alloying is also seen in H_{irr} , indicating an increase in the flux pinning by carbon alloying. An H_{irr} value of 40 T at $T=1.5$ K is obtained in alloyed fiber 2, while H_{irr} is 5.8 T at $T=25$ K for alloyed fiber 1. The H_{c2} and H_{irr} reported here are substantially higher than those previously reported for PIT MgB_2 wires ($H_{c2} \sim 37$ T in carbon-doped MgB_2 PIT wires²⁰ and $H_{irr} \sim 25.4$ T at 4.2 K in MgB_2 PIT wires with addition of SiC nanoparticles²¹). The differences may be due to the different microstructures: in carbon-doped bulk MgB_2 , the a axis shrinks but c axis remains constant with increasing carbon concentrations,²² whereas in HPCVD films both a and c axes expand with carbon alloying.¹⁴

The result here demonstrates that epitaxy and texture are not necessary for achieving high H_{c2} and H_{irr} . This is important for practical manufacturing of MgB_2 conductor wires. The high H_{c2} and H_{irr} are very attractive for superconducting magnets in magnetic resonance imaging (MRI) systems.²³ In particular, the operating temperature above 20 K allows replacement of liquid helium by efficient cryocoolers, so that MRI systems could become lighter, of lower operation cost, more reliable, and more accessible to populations in remote locations or in developing countries.^{24,25} The HPCVD technique used here is adaptable to scale up for continuous and uniform coating of long length fibers. High MgB_2 deposition rate, which is important for manufacturing, has been demonstrated up to 1.2 $\mu\text{m}/\text{min}$ by HPCVD.²⁶ Although the large diameter SiC fiber used in this work is not the desirable substrate for practical conductor wires because it is brittle and costly, MgB_2 coatings can be made on inexpensive and strong metallic wires such as stainless steel.^{10,26} Therefore,

the MgB_2 coated conductor fabricated using HPCVD could be seriously considered as a potential alternative to the current Nb-based high-field conductors.

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