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Ultra-lightweight superconducting wire based on Mg, B, Ti and Al

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Actually, MgB₂ is the lightest superconducting compound. Its connection with lightweight metals like Ti (as barrier) and Al (as outer sheath) would result in a superconducting wire with the minimal mass. However, pure Al is mechanically soft metal to be used in drawn or rolled composite wires, especially if applied for the outer sheath, where it cannot provide the required densification of the boron powder inside. This study reports on a lightweight MgB₂ wire sheathed with aluminum stabilized by nano-sized γ -Al₂O₃ particles (named HITEMAL) and protected against the reaction with magnesium by Ti diffusion barrier. Electrical and mechanical properties of single-core MgB₂/Ti/HITEMAL wire made by internal magnesium diffusion (IMD) into boron were studied at low temperatures. It was found that the ultra-lightweight MgB₂ wire exhibited high critical current densities and also tolerances to mechanical stress. This predetermines the potential use of such lightweight superconducting wires for aviation and space applications, and for powerful offshore wind generators, where reducing the mass of the system is required.

Although many superconducting elements and compounds have been discovered¹, only few of them can be used for thermally and mechanically stabilized long length wires with high current densities. Instead of high power cables and high field magnets, superconducting wires make possible the design of powerful and lightweight superconducting stators and rotors for aircraft engines and generators^{2,3}. Lightweight superconducting wires are also attractive in other specific areas, where the total mass is extremely important, e.g. powerful wind turbines^{4–6} or any space applications^{7,8}. Since the discovery of superconductivity in the lightest superconductive compound MgB₂⁹, extensive efforts have been expended in the development of practical composite wires made mostly by powder-in-tube (PIT) processes and in the enhancement of their superconductive properties, particularly the critical current density (J_c) and the upper critical field (B_c). Low cost MgB₂ superconductor wires operated at 4–25 K can lower the upfront and ongoing operational costs of superconducting systems. It was found that sheath materials play an important role in the determination of transport properties of the wires made by powder in tube (PIT) technique¹⁰. Cu is an ideal thermally stabilizing metal for low- T_c superconducting wires. In the case of MgB₂ wires, the Cu reaction with MgB₂ has to be inhibited due to a possible radical reduction of the transport current density. Therefore, a protective (i.e. diffusion) barrier has to be used (e.g. Fe, Nb or Ta) in order to avoid any reaction, namely the one between Cu and Mg¹¹. Ti sheathed MgB₂ wires were tested initially by Allesandrini¹², and Ti barriers were then successfully applied for multicore MgB₂ wires stabilized by Cu^{13,14}. Al may also be an appropriate sheath material for MgB₂ superconductor due to its high electrical and thermal conductivity, low cost, magnetism, and good formability. However, pure Al is mechanically soft metal to be used in drawn or rolled composite wires, especially if applied for the outer sheath, where it cannot provide the required densification of the boron powder. While Al alloys can offer improved mechanical properties, the conductivities and formability are markedly deteriorated. Furthermore, the solidus temperatures of Al alloys are much lower compared with the melting point of pure Al, which makes even more difficult the formation of MgB₂ by the heat treatment of Mg and B components at $\approx 650^\circ\text{C}$. The first experiment with MgB₂/Al tape superconductor was made by an *ex-situ* PIT method without final heat treatment, but it does not allow reaching high critical current density¹⁵. Several other experiments to stabilize MgB₂ wire with pure Al were also performed^{16,17}, but the stabilization was not effective enough. Also, another solution with Al bonding on the Ti sheathed wire was not successful due to intensively oxidized surfaces of both Al and Ti¹⁸. Thermally stable ultrafine-grained Al stabilized by a small content of nano-scale Al₂O₃ formed *in situ* in Al matrix, named HITEMAL (high temperature aluminum), was produced by a powder metallurgy approach^{19,20}. HITEMAL shows attractive mechanical and recently also electrical properties

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Wire	HT [°C/min]	$T_{\max}$ [°C]	Al ₃ Ti layer [μm]	HV _{0.005} [GPa]	ϵ_{irr} [%]	σ_{irr} [MPa]
wA	632/10	646.5	~1	~43	0.166	141
wB	628/10	642.5	~1	~56	0.210	172
wC	635/30	640.0	~4	~68	0.342	214

Table 1. Heat treatment conditions with initial short temperature overshoot ($T_{\max}$) of wires wA-wC and the corresponding Al₃Ti interface layer thickness, the HITEMAL sheath micro-hardness (HV_{0.005}), the irreversible strain (ϵ_{irr}) and stress (σ_{irr}).

Figure 1. The pattern of final heat-treatments applied for wires wA, wB and wC. The insert shows the resistive transitions of compared wires.

at low temperatures²¹. The first attempt to make an Al-stabilized MgB₂ wire was made using Ta diffusion barrier and HITEMAL outer sheath²², which demonstrated the possible production of Al-sheathed MgB₂ wires. It allowed to verify the utilization of Al + Al₂O₃ outer sheath for MgB₂ wire and to show what superconducting properties, especially current densities, can be reached in medium magnetic fields. Ta barrier is really heavy material (16.69 g/cm³) for lightweight composite wire, but it has been used due to minimal reaction with Al + Al₂O₃ during the final heat treatment.

In this work we present original manufacturing method and properties of ultra-lightweight superconducting wire prepared by internal Mg diffusion process (IMD) into the B, which utilizes the lightest superconducting compound (MgB₂ with 2.5 gcm⁻³) combined with the lightweight composite sheath (Al + 1.37 vol.% Al₂O₃ with a density of ~2.7 gcm⁻³) and light metallic barrier material (Ti with a density of 4.5 gcm⁻³). Due to lowered melting point of Al + 1.37 vol.% Al₂O₃ (~652 °C)²² in comparison to pure Al (660 °C) and close to melting of Mg (650 °C), really specific heat treatment is needed for MgB₂/Ti/HITEMAL wire. It should allow: (i) the fast formation of dense MgB₂ phase²³, (ii) limited Ti/Al interaction and (iii) keeping the mechanical strength of Al + Al₂O₃ sheath.

Results

It was shown that the melting point of Al + 1.37 vol.% Al₂O₃ sheath is relatively low ~652 °C²² while the temperature close to 650 °C is required for the fast formation of a dense MgB₂ phase²³. This could cause undesirable changes (e.g., melting or recrystallization) of the Al₂O₃ stabilized Al sheath. Therefore, fast ramp heat treatments (~25 °C/min) with the setting temperature 628–635 °C and overshoot up to 640–646.5 °C were applied for as-deformed Mg/B/Ti/HITEMAL wires named as wA, wB and wC, see Table 1 and Fig. 1. A transversal cross-section image of the wB wire is shown in Fig. 2(a), where the central hole (at the place of the original Mg core) and formed MgB₂ layer of thickness ~100 μm are well visible. It correlates with the kinetic of MgB₂ layer formation presented by Li et al. who have calculated the time needed for the Mg + B reaction²⁴. Our previous experiments have confirmed this model and showed the optimal time of 8 minutes for HT at 635 °C and overshoot 654 °C²³. Figure 2(b) shows a thin intermetallic reaction layer with a thickness of ~1 μm at the Ti/Al interface of the wB wire subjected to heat treatment (HT) temperature 628 °C/10 min.

The local EDS analysis confirmed Al₃Ti phase, which is in good agreement with other studies²⁵. A comparable Al₃Ti layer of a similar thickness was observed for the wA and wB wires that were heat treated for 10 min, while for the wC wire that was annealed for 30 min, the layer increased to ~4 μm (see Table 1). Formation of the Al₃Ti phase at the Ti/Al interface may significantly decrease thermal and electrical transport between the MgB₂ core and outer Al + Al₂O₃ sheath. To minimize the Al₃Ti phase formation, a short heat treatment regime with a very fast initial ramp is preferred.

The critical current densities of the compared wires wA-wC were determined from the magnetic loops by using Bean's critical state model to establish a relationship between the width of the hysteresis loop Δm and the critical current density. Assuming a full penetration of the measured sample by a magnetic field, the particular

Figure 2. Transversal cross-section image of a heat-treated wB wire (a) and a detail of an Al₃Ti reaction layer at the Ti/Al interface of the wB wire (b).

Figure 3. Critical current densities of the wB wire obtained from the VSM measurement (a) and $J_e(B)$ from the transport DC measurement of wires wA, wB and wC at 4.2 K (b) including J_e of wB wire from VSM measurement.

form of the formulae relating Δm to J_c can be derived with regard to the current flow geometry. In the case of a cylindrical MgB₂ core, the critical current density is obtained according to:

$$J_c = \frac{3}{d} \Delta M \text{ for } B \text{ applied parallel to the wire axis } (B \parallel) \text{ and}$$

$$J_c = \frac{3\pi}{4d} \Delta M \text{ for } B \text{ applied perpendicular to the wire axis } (B \perp)$$

where ΔM is the width of the hysteresis loop divided by the volume of the MgB₂ core, and d is the core diameter. For the wires made by the IMD process resulting in an annular MgB₂ core shown in Fig. 1(a), all formulae must be multiplied by a factor considering the hollow core geometry²⁶. The factor is $\left(1 - \frac{d_i^2}{d_o^2}\right) / \left(1 - \frac{d_i^3}{d_o^3}\right)$, where d_i and d_o is the inner and outer diameter of the MgB₂ core and the formula for J_c is:

$$J_c = \frac{3\Delta M}{d_o} \frac{1 - \frac{d_i^2}{d_o^2}}{1 - \frac{d_i^3}{d_o^3}}$$

Filament dimensions d_i and d_o in wB wire are 0.00423 and 0.00625 cm, respectively. Figure 3(a) shows the critical current densities of the wB wire measured by a vibrating sample magnetometer at external magnetic fields 1–9 T (in $B \parallel$ and $B \perp$) and temperatures 5–25 K. $J_c(B, T)$ values of the wire in the perpendicular field are identical with J_c of a single core MgB₂/Ti/Cu wire made by the IMD process and annealed at 640 °C/60 min²⁷, which is a result of a sufficiently dense boron powder deformed inside the Al + Al₂O₃ sheath. The dotted lines present J_c values at a parallel field, which are very similar to the perpendicular one for temperatures 5–15 K, and only slightly lowered at temperatures above 20 K. A small J_c difference between the perpendicular field direction (with currents flowing

Figure 4. Strain tolerances of the wA, wB and wC wires to tensile stress at 4.2 K compared with a similar wire with a GlidCop outer sheath.

along the tubular core) and parallel one reflects an excellent homogeneity of the MgB_2 compound created by the IMD process. An opposite behavior with large J_c differences between $B\parallel$ and $B\perp$ was observed in MgB_2 made by *in-situ* PIT process, which was attributed primarily to a texture caused by wire deformation and resulting to different ‘porosity’ or ‘connectivity’ in longitudinal and transversal direction²⁸. Figure 3(b) shows the transport engineering current densities (calculated to the whole cross-section of the wire) at 4.2 K for wA, wB and wC samples. The highest J_c is measured for the wA due to the $T_{\text{max}} = 646.5^\circ\text{C}$, which is close to the melting point of Mg ²². The mechanism of presented IMD process at temperatures below 650°C considers fast Mg diffusion into boron powder and subsequent creation of MgB_2 phase^{23,24}. The resistive transitions of compared MgB_2 layers are similar (see the insert in Fig. 1), but the systematic decrease of critical temperature ($T_c = 37.00\text{ K}$ - wA, 36.89 K - wB and 36.87 K - wC) and small widening of $R(T)$ transition ($\Delta T_c = 1.90\text{ K}$ - wA, 2.03 K - wB and 2.15 K - wC) can be observed. It reflects the composition and purity of created MgB_2 phase and correlates with J_c values shown by Fig. 3(b).

Only slightly lowered peak temperature of 642.5°C for the wB wire resulted in around 10% lower J_c compared with that of the wA wire, but J_c of wC wire is lowered by 37% at field 6 T in comparison to wA. Figure 3(b) shows also the relation between the transport and magnetic J_c (from VSM), for the wB wire, where a falling off of the magnetic J_c from the transport one was observed. It can be rationalized considering a different current flow combined with no fully identical connectivity along and across the core axis.

Changes in the critical currents of present wires subjected to axial tension at 4.2 K are shown in Fig. 4. Due to a larger thermal contraction of Ti and Al compared with that of MgB_2 , cooling down to 4.2 K results in compression stress which acts on the MgB_2 layer and reduces the critical temperature and current^{1,29}. Applied axial tension partially compensates the pressure stress and consequently critical current increases up to a level of irreversible strain (ε_{irr}), where the breaking of brittle MgB_2 leads to a radical degradation. The ε_{irr} value defines the maximum strain at which the current still remains reversible²⁹. One can see a considerable effect of applied heat treatment on the irreversible strain in Figure 4. The wA wire with the highest peak temperature of 646.5°C behaves mechanically as the weakest, and consequently the lowest $\varepsilon_{\text{irr}} = 0.166\%$ is measured due to the apparent softening of the outer sheath.

However, the wC wire annealed at the peak temperature of 640°C has the highest $\varepsilon_{\text{irr}} = 0.342\%$, which is even comparable with the strain limit of a single-core IMD wire reinforced with a GlidCop sheath, see the filled circles in Fig. 4. GlidCop is dispersion strengthened copper which was already effectively used for some MgB_2 wires²³. Table 1 shows the irreversible strain ε_{irr} and irreversible stress σ_{irr} measured for wA-wC, which are correlating well with the sheath microhardness $\text{HV}_{0.005}$ – the lowest for wA $\sim 43\text{ GPa}$ and the highest for wC $\sim 68\text{ GPa}$. It was already shown that strain and stress tolerances (σ_{irr} and ε_{irr}) of MgB_2 wires are dominantly affected by mechanical strength of the outer sheath³⁰. Therefore, structural changes in outer sheath of wires wA-wC were examined by transmission electron microscopy.

The as-deformed $\text{Al} + \text{Al}_2\text{O}_3$ consists of Al grains intensively elongated in the wire drawing direction and transversal structure shows a randomly distributed nanometric Al_2O_3 dispersoids, see Fig. 5(a). The nano-dispersoids stemmed from native amorphous (am)- Al_2O_3 layers on as-atomised Al powders²⁰. The induced shear deformation broke up the Al_2O_3 layers into am- Al_2O_3 platelets during the cold working steps²¹, and some remnants of the fractured am- Al_2O_3 platelets remained at high angle grain boundaries, see the white arrow in Fig. 5(a). However, a majority of the am- Al_2O_3 platelets transformed into nanometric crystalline Al_2O_3 dispersoids during the cold working were found at both, the Al grain boundaries and within the Al grain interiors, see the black arrow in Fig. 5(a). During the final heat treatment Al grains coarsening is observed. High angle grain boundaries are preferentially eliminated with increasing temperature, but low angle grain boundaries are still stabilized by Al_2O_3 dispersoids and are sustained even higher annealing temperatures, see Fig. 5(b). The black arrows show co-localization of low angle grain boundaries with Al_2O_3 dispersoids in the wA wire sheath.

Figure 5. TEM bright field images of transversal-sections of the as-deformed Al + Al₂O₃ sheaths (a) and the heat-treated sheath of wA wire (b).

Figure 6. TEM bright field images of transversal-sections of the Al + Al₂O₃ sheaths of the wA (a), wB (b), wC (c) wires and of the as-deformed one (d).

Figure 6 shows TEM bright field images of transversal sections of the Al + Al₂O₃ sheath in the heat-treated wires wA - wC (a-c) in comparison to as-deformed one shown by Fig. 6(d). The TEM micrographs demonstrate the different microstructure upon annealing with the peak temperature between 640°C and 646.5°C. While Al

grains of as-deformed wire have generally equiangular shape of averaged size $d_{av} \sim 470$ nm, enlarged and/or elongated (not equiangular) grains are visible in wB and wC wires due to grains coarsening. High angle grain boundaries of Al + Al₂O₃ sheaths are yet well stabilized by Al₂O₃ dispersoids at heat treatment temperature $T_{max} = 640$ °C, see Fig. 6(c), where nearly doubled grain size $d_{av} \sim 950$ nm in comparison to as-deformed sheath is found. The outer sheath of wC wire stays polycrystalline with the structure similar to the as-deformed one, see Fig. 6(d). The grain size structure of wB sheath shown by Fig. 6(b) is more affected by annealing only ~ 10 °C below the melting of Al + Al₂O₃ and $d_{av} \sim 1380$ nm was estimated for $T_{max} = 642.5$ °C. Figure 6(a) shows that grain boundaries in wA (5.5 °C below the melting of Al + Al₂O₃) are not more stabilized and a big Al grains with sub-grains and low angle grain boundaries with localized Al₂O₃ dispersoids are present. Consequently, the correct estimation of d_{av} for wA wire is not possible. Observed structural changes and grains coarsening leads to mechanical softening of heat treated Al + Al₂O₃ sheaths, which is accompanied by the decreased sheath micro-hardness (see Table 1) in comparison to not annealed Al + Al₂O₃ wire with $HV_{0.005} \sim 70$ GPa²².

Discussion

The presented microstructural study clearly illustrates that the different Al grain structure of the Al + Al₂O₃ sheaths strongly affects the wire responses to axial tension. The Al + Al₂O₃ is a suitable material for a sufficiently strong superconducting wire, but conditions of the final heat treatment have to be controlled very precisely. Figure 4 shows the different strain tolerances, which are strongly affected by the applied annealing influencing the sheath microstructure (see Figs 5 and 6). While the apparent critical current degradation in the wA wire occurred at the tensile stress of 141 MPa due to not more stabilized grain boundaries by Al₂O₃ dispersoids, the wC wire is able to withstand much higher stress of 214 MPa. Due to polycrystalline structure and grain size $d_{av} \sim 950$ nm, the mechanical strength of the wC wire was by $\sim 25\%$ higher than determined for the wA wire, which is characterized by big grains with sub-grains and low angle grain boundaries. The averaged grain size of wB sheath ($T_{max} = 642.5$ °C) is ~ 1380 nm, which is larger than for wC and consequently $\sigma_{irr} = 172$ MPa is measured, see Table 1. Similar correlation (sheath softening) is observed by the micro-hardness ($HV_{0.005}$) data, which decreased from $HV_{0.005} = 68$ to 43 as the peak temperature increased from 640 to 646.5 °C, respectively (see Table 1). Nevertheless, the achieved $HV_{0.005} = 43$ for the Al-Al₂O₃ sheath of the wA wire remains still much higher than that of pure Al ($HV_{0.005} = 27$)²¹ due to a dense net of low angle grain boundaries effectively stabilized by Al₂O₃ dispersoids. The observed differences are attributed only to structural changes in the Al + Al₂O₃ material (and formation of thicker Al₃Ti layer) subjected to different heat treatment. Therefore, precisely chosen heat treatment has to be applied to form a high current density MgB₂ core along with a high strength Al + Al₂O₃ sheath and Ti diffusion barrier with a limited interfacial reaction at the sheath interface.

Calculation of conductor mass based on the MgB₂/Ti/Al + Al₂O₃ can lead to at least 2.5 times weight reduction when compared with a typical MgB₂/Nb/Cu wire of the same cross-sectional dimensions. This clearly outlines the potential of the lightest MgB₂/Ti/Al + Al₂O₃ superconducting wire, when compared with any other metallic or ceramic superconductors. Consequently, presented MgB₂ wire meets demanding requirements on electrical and mechanical properties of superconductors for efficient superconductive and light-weight applications.

Methods

A single-core MgB₂ wire was fabricated by internal magnesium diffusion (IMD) into a boron process. Pure Mg99.99% rod 2.9 mm in diameter was precisely positioned in the central axis of a Ti99.99% tube with 5.5 mm inner diameter and 7.2 mm outer diameter. The free volume between the Ti tube and Mg rod was filled by a B99.8% powder (<1 μ m size) in a glove-box under pure Argon atmosphere. The Mg/B/Ti composite was rotary swaged down to 6.2 mm diameter, cleaned, and inserted into a HITEMAL tube with 6.3 mm inner diameter and 9.1 mm outer diameter, that was machined from an as-extruded Al + 1.37 vol.% Al₂O₃ rod²¹. The Mg/B/Ti/Al + Al₂O₃ composite rod was rotary swaged down to 7.5 mm and then groove rolled to a rectangular wire with a cross-section of 1.02×1.02 mm². A heat treatment process was applied at 300 °C for 30 min during the groove rolling process each time after reaching around 50% area reduction. The volume composition of the as-deformed wires corresponded to around 11% Mg, 12% B, 27% Ti, and 50% Al + Al₂O₃ outer sheath. The following final heat treatment was applied under Ar atmosphere at: (i) 632 °C for 10 min (wA wire); (ii) 628 °C for 10 min (wB wire); and (iii) 635 °C for 30 min (wC wire), with the peak temperatures of 646.5, 641.5 and 640 °C, respectively, see Fig. 1 and Table 1.

Hysteresis loops measured by a vibrating sample magnetometer (VSM) option in PPMS of Quantum Design system were recorded between -2 and $+9$ T with a constant field sweep of 6.3 mT/s in a temperature range of 5–25 K (at 5 K steps), and the field directed perpendicular and parallel to the wire axis. Using Bean's critical state model, the critical current density J_{c-mag} was determined. Resistive transitions were measured by a standard four-probe method with DC current magnitude of 100 mA. Critical temperature (T_c) and the width of transition (ΔT_c) were determined from $R(T)$ dependences shown by the insert in Fig. 1. Transport critical currents were measured at liquid He temperature and an external magnetic field from 4.0 to 8.0 T using standard DC measurement with $1 \mu V cm^{-1}$ criterion for I_c values. A free-standing short sample (~ 50 mm) configuration was used for the tensile load tests of the wires at 4.2 K²⁹. The electro-mechanical characteristics: I_c versus the tensile strain (ϵ) and the stress-strain curves $\sigma(\epsilon)$ were measured at a constant external magnetic field of 6 T. Scanning electron microscopy (SEM, JOEL 7600 F) with energy dispersive spectrometry (EDS, Oxford Instruments X-Max 50) was used to characterize polished transversal-sections of the heat-treated wires. Transmission electron microscopy (TEM) observations were made using JEOL JEM 1200FX microscope. TEM specimens were prepared by mechanical grinding and polishing followed by Ar beam ion milling using GATAN PIPS II. The transversal Al grain size (d_{av}) was determined by image analyse of multiple bright filed TEM micrographs.

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Author Contributions

P.K. and I.H. participated in technological verification. P.K. prepared the manuscript and Figures 2–3. A.R. performed SEM and TEM microscopy analyses and prepared Figures 1, 4–5. M.K., J.K., T.M. and L.K. performed electrical and mechanical characterization at low temperatures. M.B. and P.Kr. fabricated extruded Al + Al_2O_3 material for the outer sheath. All authors reviewed and approved the final manuscript.

Additional Information

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