Composite Superconducting MgB_2 Wires Made by Continuous Process

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Abstract—Previously developed manufacturing technology of a low-cost composite single core based MgB_2 superconductive wires has been investigated in details using monel sheath and titanium diffusion barrier. In this process, Mg and B as well as nanoSiC dopant powders were fed continuously to a “U”-shaped metallic sheath titanium, and subsequently monel sheath. Resulting wires were assembled in a “6 + 1” configuration cable constructed from six 0.75-mm Monel/Titanium/(MgB_2 + 10 wt%SiC) and Monel/Titanium/ MgB_2 wires and one central copper wire with two twist pitch arrangements: 13 mm. Undoped and SiC-doped wires were compared in respect of their $I_c(B, T)$ characteristic. Wires were sintered at 700 °C for different times and subsequently were initially tested in a dedicated helium force vapor cooling system at temperatures 20–35 K at 1 T. Systematic and statistic $I_c(B, T)$ measurements of the wires and cables in LHe were conducted at high external magnetic flux density up to 9 T. It was shown that wire feeding, sealing, reduction, and forming technology enable virtually unlimited conductor lengths as well as in-line processing control simultaneously ensuring a high degree of reproducibility and consistent quality of the MgB_2 superconductor. Such generic single core MgB_2 conductors enable manufacture of multifilamentary MgB_2 conductors with desired configurations (e.g., twist pitch for transient losses and ac applications).

Index Terms— MgB_2 wires, superconducting cable, twisting, critical current, MRI, direct current (DC), gaseous helium cooling, continuous process.

I. INTRODUCTION

AFTER introduction of *in situ* and *ex situ* formation of MgB_2 Powder-in-Tube (PIT) conductor in 2001 [1] there is an urgent need for cost effective manufacture and large quantities of MgB_2 superconductors for a variety of applications working at the temperatures range of 15–20 K [2], [3]. Such a need puts a real pressure on the manufacturing capabilities of

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Fig. 1. Schematic illustration of the manufacturing technology stages: (a) single core conductor: S1 - bimetallic sheath dispenser, S2 - bimetallic tape “U” profiling, S3 - mixed powder delivery system into “U” profiled tape, S4 - powder thermal pre-conditioning, S5 - bimetallic tape closure and laser welding, S6 - wire reduction, S7 - eddy current control; (b) Twisted multifilamentary MgB_2 conductor formation in protective gas atmosphere.

the traditional PIT batch technology of multifilamentary MgB_2 wires [4].

Introduced in 2016, a novel process of continuous manufacture of *in situ* MgB_2 wires brought a new dimension to production of the large-scale superconducting MgB_2 wires capable to potentially replace NbTi conductors for MRI applications [5]. Doping of the *in situ* MgB_2 with SiC nano-powder improves $I_c(B, T)$ properties at magnetic flux densities above 2–4 T. The actual position of $I_c(B, T)$ ‘cross over’ between doped and undoped wires is a function of many materials-processing related factors specific for a particular manufacturing technique.

II. MANUFACTURING PROCESS

A. Principles of the Technology

Fig. 1(a) schematically illustrates the manufacturing technology stages of a single core conductor. Proposed process of has been in details described in earlier publication [4] and has some similarities with continuous tube filling and forming (CTFF) process [6], [7].

The welded Monel/Titanium/(MgB_2 + 10 wt%SiC) and Monel/Titanium/ MgB_2 wires after stage S5, Fig. 1, were then passed through a series of rollers and dies, stage S6, to reduce the diameter down to 0.75 mm. Such a single core MgB_2 wire is then subjected to a bundling process (e.g., a central copper wire and 6 single core MgB_2 wires in a “6 + 1” configuration) with

Fig. 2. Boron particle size analysis using Mastersizer 2000.

Fig. 3. (a) Photograph of a “6 + 1” configuration cable constructed from six 0.75 mm Monel/Titanium/(MgB₂ + 10 wt%SiC) wires and one central copper wire showing twist pitch 13 mm and 26 mm; b) cross section of the “6 + 1” unreacted Monel/Titanium/(MgB₂ + 10 wt%SiC)/Cu conductor.

desired twist pitch and sequentially on-line heat-treated under protective gas atmosphere as presented in, Fig. 1(b).

B. Powder Preparation and Feeding

In situ MgB₂ wires were prepared using amorphous boron from Pavzeyum, Turkey, magnesium from Magnesium Metal Co., Turkey and SiC dopant from Iolitec, Germany. Amorphous boron has a purity of 95–97% with a particle size distribution presented in Fig. 2, magnesium has a purity of above 99.9% with a particle size of 100–150 μm and nano SiC has a particle size of 40–60 nm. During the mixing process oxygen, hydrogen and moisture levels were monitored. For the wires with SiC doping, doping level was fixed to 10 wt%. The mixed powders are introduced into a “U” profile of titanium/monel sheath with liquid carrier.

C. Wire Size Reduction and Twisting of Wires

After laser welding (see Fig. 1(a)) stage S5) wire is passed through a series of rollers and dies, which reduces the diameter down to 0.75 mm. Total elongation of wire from the laser-seam welding station to 0.75 mm diameter is equivalent to 98.5% reduction in area, resulting in heavily compacted and densified wires (Fig. 3).

Six individual long length single core MgB₂ conductors (undoped or doped with SiC), together with a central copper wire, were introduced to a wire twisting machine to produce a “6 + 1” multifilamentary cable configuration, with twist pitches of 13 mm and 26 mm, Fig. 3.

Fig. 4. Polarized reflective optical cross-sectional images of the MgB₂ + 10 wt% SiC Titanium/Monel single conductor core of the wire, see Fig. 2(b), sintered at 700 °C for different time under argon protective atmosphere: (a) 0 min, (brown areas represent agglomerates of amorphous boron powder, where bright areas represent Mg and SiC); (b) 1 min, (it is evident that reaction of the Mg diffusion is very rapid); (c) 2 min; (d) 3 min, there is no strong evidence of the unreacted Mg zones however unreacted Mg pockets remain in the cross section; (e) 10 min (one may notice developed difference in the reflective light spectrum in relation to samples annealed at shorter times); (f) 30 min (reflective polarized image after 60 min sintering looks identical as for wire annealed after 30 min). (Pictures e) and (f) show some amount of blue-like color inclusions, which is associated with formation of Mg₂Si as presented in Fig. 6).

Fig. 5. SEM image of the central core of a 0.75 mm Monel/Titanium sheathed MgB₂ wires sintered at 700 °C for 30 min: (a) MgB₂, (b) MgB₂ + 10 wt% SiC showing white Mg₂Si inclusions, the black areas represent porosity. The bar at the bottom of images represents 100 μm distance. Image analysis of the white inclusions population in Fig. 4(f) is almost the same as in Fig. 5(b).

E. Heat Treatment and Bundle Consolidation and Insulation

In situ technique adopted in presented production of the long lengths of the superconductive MgB₂ wires with relatively narrow band particle distribution of boron and magnesium enable formation of MgB₂ superconducting compound in a relatively short time, therefore an in-line thermal treatment process can be adopted rather than the usual batch heat treatment process. The process used in the current production was optimized for 30 minutes sintering at the desired sintering temperature, which in presented case was chosen to be 700 °C. The electrical insulation will be introduced in the later stage of the production according to requirements of the potential customers and applications. Microstructural analysis of the reactivity of the used powders supported by DTA analysis prove that the completion of the conversion takes place in first 5–10 minutes at 700 °C as demonstrated in Fig. 4. It is noticeable in Fig. 4 that the gray-brown reflective image of the superconductive core after

Fig. 6. EDX weight % compositional analysis of the central core of a 0.75 mm Monel/Titanium sheathed MgB_2 + 10 wt% SiC wire sintered at 700 °C for 30 min. The measured values have an accuracy of 0.1 wt%. At marked *cross analysis point*, weight composition of the white extended inclusion was 61.8 wt% Mg and 38.3 wt% Si where calculated values for Mg_2Si are 63.4 wt% Mg and 36.6 wt% Si. Clearly indicating on formation of most stable Mg_2Si impurity phase in the Mg-Si system [8].

30 min sintering becomes more blue-like, indicating development of the properly ordered MgB_2 structure and reflectivity of Mg_2Si residual phase formed in case of the doped sample, Fig. 6. Critical current measurements of the doped wire after 10 min and 30 min sintering show very similar values as it would be presented in next paragraph.

III. CRITICAL CURRENT MEASUREMENTS

A. Helium Force Vapor Cooled Critical Current Testing

The critical current of the Monel/Ti sheathed wires was measured using a purpose designed and build helium force vapor cooled critical current testing facility [9]. The required temperature is achieved via two heaters, one of which is controlling the evaporation rate of helium and thus the vapor flow rate, the second heater is used to adjust the vapor temperature to the required value. Sample temperature was measured directly below sample voltage contacts via a carbon ceramic sensor. A uniform magnetic flux density in the range from 0 to 1 T was applied over a 5 cm region across the center of the sample. Sample voltage contacts were placed 1 cm apart. Wire lengths of 15 cm were used for testing to ensure that all current is transferred from the sheath to the superconducting core. Current contacts were 4 cm long. An electric field criterion of $E = 1 \mu\text{V}/\text{cm}$ was used.

Critical current measurements of the doped wire after 10 min and 30 min sintering show very similar values see Fig. 7 providing evidence for on-line sintering possibilities for the wires produced, see Fig. 1(b).

B. Helium Cooled High Magnetic Field Critical Current Measurements of Wires and Bundles

Critical currents of the single core SiC doped and undoped wires sintered for 30 min at 700 °C in argon protective atmosphere were fully characterized in range of temperatures in external flux density up to 9 T are presented in Figs. 8 and Fig. 9 respectively.

A cable sample of the six MgB_2 monofilaments around one copper wire was wound around a TiAlV barrel with a spiral groove and sintered for 30 min at 700 °C in argon protective

Fig. 7. Comparison of critical current values of Monel/Titanium sheathed MgB_2 + 10 wt% SiC 0.75 mm wires versus temperature after sintering at 700 °C for 10 and 30 min under magnetic field of 1 Tesla. It is noticeable that values for 10 min are slightly higher than for longer time.

Fig. 8. Transport critical current measurements I_c of a single core undoped sheathed MgB_2 conductor. Total length of 3, 5 km was split in 7 pcs of length 0, 5 km. At each 0, 5 km, 2 samples were taken, resulting in a group of 2×6 samples (ends were not taken). 4 random samples within these 12 samples were tested and represented above. The diameter of the wire is 0.75 mm and cross section of the superconducting core is equal 0.1370 mm^2 corresponding to 31% fill factor.

Fig. 9. Transport critical current measurements I_c of a single core doped sheathed MgB_2 conductors measured at 4.2 K. Total length of 9,1 km single core conductor was split in 7 pcs of length 1, 3 km. At each 1, 3 km, 2 samples were taken, resulting in a group of 2×6 samples (ends were not taken). All 12 samples were tested and presented in graph. The diameter of the wire is 0.75 mm and cross section of the superconducting core is equal to 0.1370 mm^2 corresponding to 31% fill factor.

atmosphere. Both ends were subsequently soldered to copper discs for connection to the current feeds and the sample was fixed to the barrel using epoxy. Sample voltage contacts were placed in the middle of the 2.6 m long sample connecting all wires individually along a single twist pitch length. A uniform magnetic flux density in the range from 3 T to 6 T was applied over the entire length of the sample such that the Lorentz force is pointing inward.

Fig. 10. (a) Picture of the spool of the single core $\text{MgB}_2 + 10 \text{ wt\% SiC}$ Titanium/Monel conductor; (b) Image of sintered and tested “6 + 1” Monel/Titanium/($\text{MgB}_2 + 10 \text{ wt\%SiC}$)/Cu conductor as presented in Fig. 2(b). Conductor sample length 2.6 m; twist pitch 13 mm, diameter of the single core conductor wire 0.75 mm, sintering temperature 700 °C using Wind and React method to avoid influence of the bending procedure on the small radius. Position of the six voltage taps (V1–V6) is also visible in the centre of the picture.

TABLE I
CRITICAL CURRENT, I_c AND n -VALUES MEASURED AT 3 T, 4 T AND 5 T AND AT 4.2 K FOR DOPED CONDUCTOR, 13 MM TWIST PITCH

	3T		4T		5T	
Voltage taps	I_c (A)	n	I_c (A)	n	I_c (A)	n
V1	-	-	415	47.9	229	37.9
V2	-	-	412	49.2	228	38
V3	643	23.6	377	19.5	214	20
V4	649	19.8	386	20.8	218	21.6
V5	698	57.1	405	41.8	223	30.3
V6	697	49.2	408	43.4	229	40.6
‘cabled 1 filament’, $I_c/6$	111.96		66.75		37.25	
‘not cabled 1 filament’	112.1		65		37.2	
I_c ratio of ‘cabled’ to ‘not cabled 1 filament’	1		1.03		1	

The voltage current traces of all six single-core MgB_2 wires were measured individually, while all single core MgB_2 wires were carrying current in parallel, in order to detect possible current non-uniformity issues. The critical current, I_c , is defined at a criterion of $E = 10 \mu\text{V/m}$ and the n -value (representing the steepness of the voltage-current characteristic) is taken in the range from 10 to 100 $\mu\text{V/m}$. Six sampling voltage contacts, from V1 to V6, (see Fig. 10 and Table I) were placed in the middle of the cable sample, 1 meter apart, on each of the 6 filaments individually along twist pitch length. Transition curves and resulting critical current values $I_c/6$ of the cabled individual filaments, ‘cabled 1 filament’ (Table I), were measured and compared to single filament average critical current values of the same batch (see Fig. 9) sintered but not cabled ‘Not cabled 1 filament’ (Table I). Measurements conducted on the individual single core conductors of the twisted “6 + 1” conductors in configuration presented in Fig. 10(b) as well as average value of the single not cabled wire are summarized in Table I. The cross over between the SiC doped and undoped samples takes place at $\sim 4 \text{ T}$ for 5 K indicating applicability of both conductors for standard 1.5 T MRI magnets.

It is evident that all individual wires are characterized by almost the same critical parameters and the values of the 13 mm twist pitch are unaffected by the adopted continuous twisting process used at the industrial environment.

It has been shown that in case of the Monel/Titanium sheathed ($\text{MgB}_2 + 10 \text{ wt\% SiC}$) wires diameter 0.75 mm in “6 + 1” conductor, there is no degradation of the I_c values of the individual untwisted reacted wires in comparison to twisted and reacted cable.

IV. CONCLUSIONS

It is documented that a novel manufacturing technology to produce infinitely long and cost effective composite single core MgB_2 superconductive wires that effectively can be twisted to “6 + 1” conductor configuration with twist pitch 13 mm.

The critical current density after twisting proves to be identical to the critical current density of a single core conductor. Development of alternative sheath materials such as mild steel and austenitic stainless steel are in progress to enable cost reduction without degradation of superconductive and mechanical properties. There is also work on improved cryostability of the wires in “6 + 1” conductor which will be reported in the near future.

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