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Advances in Development of Powder-in-Tube Nb₃Sn, Bi-Based, and MgB₂ Superconducting Conductors

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Analysis of the applicability of the low cost powder-in-tube technology of technical Nb₃Sn, Bi₂Sr₂CaCu₂O_{8+d} and MgB₂ superconducting conductors has been presented in respect of their complexity in emerging hydrogen cryomagnetic technology where temperature of the liquid hydrogen can be as low as 14.1 K, and all above conductors can be considered having a comparable $J_c(B, T)$ characteristics. The new emerging hydrogen economy where liquid hydrogen can serve as an energy carrier and cryogenic coolant with exceptionally high latent heat value presents opportunities for the range of superconducting materials characterised by T_c applicable at hydrogen technologies. All above powder-in-tube conductors are exploring solid-liquid reactive diffusion processes originated from the complex compounds or intermetallics, defining the actual “technical price” in (€/kA m) of the resulting wire compound based on $J_c(B, T)$ characteristics and materials as well as manufacturing cost and complexity. This rather complex techno-economic aspect of superconducting powder-in-tube conductors needs to be addressed and analysed to help to solve a trilemma concerning powder-in-tube conductors at low temperatures.

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1. Introduction

The discovery of high temperature superconducting materials, HTS, and medium temperature superconducting material, MTS, (MgB₂), in 1986 and 2001 correspondingly and research on round powder-in-tube (PIT) conductors does not eliminate the old well-established Nb-based A-15 superconductors. In contrary, lack of the reliable strong, long lengths, high current round conductors operating at high magnetic field brought a renaissance to the Nb-based, mostly Nb₃Sn PIT [1] and restacked-rod processed (RRP) [2] technologies for large-scale applications in 21st century.

As the application of high specification MgB₂ and Bi₂Sr₂CaCu₂O_{8+d} superconductors gradually becomes a reality it is clear that incremental progress in the development of all superconducting PIT materials is the key to success of superconductivity applications at hydrogen temperatures in range of 14 ÷ 20 K. Superconducting materials can only be applied according to an engineering specification that has to be determined for each particular application from the design requirements. Economic viability and operation and safety margins in service need to be taken into account as well. In this article, we will analyse three main PIT conductors Nb₃Sn, MgB₂, and Bi₂Sr₂CaCu₂O_{8+d} with potential of the implementation. Is our intention to bring discussion about priorities to be given cool most promising superconducting

PIT conductor. Nb₃Sn is characterised by lowest $T_c \approx 18$ K but it is the most technologically established among three of them. Bi₂Sr₂CaCu₂O_{8+d} has the highest critical temperature but $B(T)$ reversibility dependence makes this material useful below LH₂ temperatures.

All three discussed above PIT conductors are exploring solid-liquid reactive diffusion processes originating from the complex compounds or intermetallics. One of the mayor drawback concerning resulting PIT wires is that the final superconducting core density cannot be reached due to or retrograde densification as it is in case of Ag–Bi₂Sr₂CaCu₂O_{8+d} or phase density difference due to phase transformation as it is for MgB₂ and Nb₃Sn. The only realistic approach to achieve the dense highest quality superconducting core is to develop a reliable kinetic *in situ* thermo-mechanical densification technology. In this article, the evolution of research activity will be followed from near market industry driven design and development of fully engineered conductors through to research on basic and enabling science for materials processing that is largely academic and curiosity driven. The most effective path to application depends on a considered balance of research that is different for each conductor family depending on the state of maturity of the conductor processing route. As a consequence, the type of research activity appropriate for the development and optimisation of a particular “conductor of choice” processing route varies depending on the maturity of the technology as well. But also we need to bear in mind that emerging hydrogen cryomagnetic technologies will require operation of the PIT superconductors at 14 ÷ 20 K range [3].

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2. Nb₃Sn powder-in tube multifilament conductor

Historically, the first PIT conductors were manufactured using variety of combinations of Cu and Nb powder mixture which was subsequently infiltrated or interdiffused by Sn to form Nb₃Sn conductors [4]. These processes however scientifically interesting are complex and very difficult to scale up.

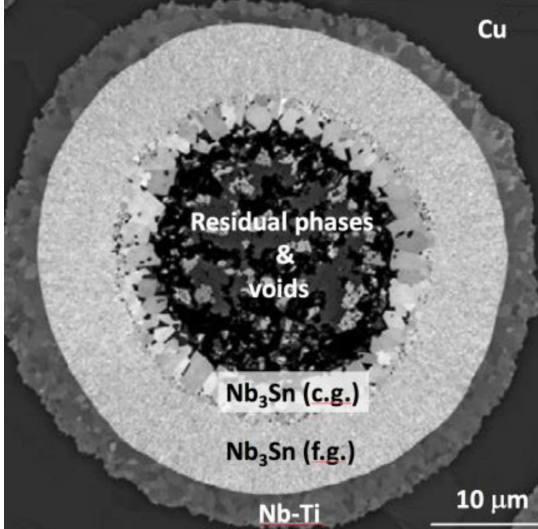


Fig. 1. Transverse cross-section of a single tubular filament after reaction of a 192-filamentary PIT Nb₃Sn conductor manufactured by Bruker EAS. In the centre, marked as “residual phases” there is a core made of large Nb₃Sn grains (light contrast), Cu-rich phases (dark contrast) and large irregular voids (black). The “Nb₃Sn (c.g.)” represents layer of coarse grains of Nb₃Sn followed radially by a thick annulus of fine grain Nb₃Sn marked as “Nb₃Sn (f.g.)”. The grey ring represents the residual Nb7.5 wt%Ta tube which acts as also residual diffusion barrier between intermetallic superconducting layer and Cu stabilising matrix. One may realise that the only “Nb₃Sn (f.g.)” ring is conducting an effective critical current. Adopted after Tarantini et al. [5].

Recent years an intensive research on PIT Nb₃Sn conductors using initial powder mixture of NbSn₂, Sn and Cu inserted in Cu-clad Nb7.5 wt%Ta tube where the powder was separated from the tube by a thin Cu sleeve results in exceptionally high J_c of a very fine Nb₃Sn layer formed [5], see Fig. 1. Process is rather very complex where 4-stage heat treatment is used to provide the finest structure of Nb₃Sn conductor. Such a multistage complex heat treatment of intermetallic NbSn₂ powder in presence of Cu, Sn and Nb results in formation of the range of intermediated phases such as: Cu₆Nb₅, Cu₃Sn, Nb₆Sn₅ before the final Nb₃Sn layer is formed [5].

However, a substantial improvement of $J_c(B)$ at 4.2 K of state of the art powder-in-tube niobium-tin superconductors is well documented, see Fig. 2 [1], but there are three major draw backs: *firstly*, there is no change in

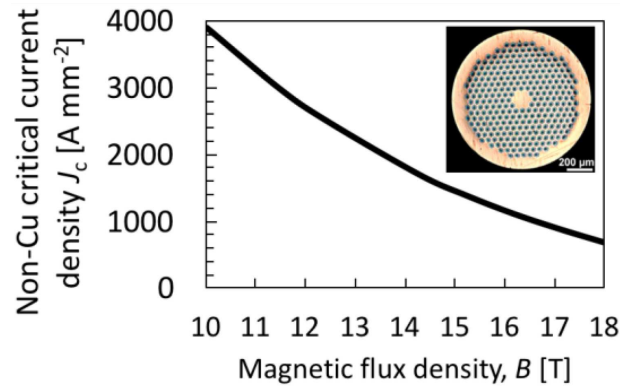


Fig. 2. Non-Cu critical current density as function of magnetic flux density at 4.2 K for 288 filament PIT wire diameter 1.255 mm, manufactured by ShapeMetal Innovation. After Godeke et al. [1]. Individual filaments are approximately 35 mm in diameter, where Cu constitutes for 55% of the overall cross-section of the wire. Non-copper data is specifically used in comparison of the performance of the actual superconducting portion of “Nb₃Sn conductors” due to wide variation of the superconductor to Cu-cryostabiliser ratio used by industrial manufacturers. The presented non-Cu J_c value corresponds to I_c divided by the sum of surfaces of cross-section of the total number of individual filaments. In case of the engineering critical current density, J_{ceng} , presented in plot critical current values will be reduced by $\approx 55\%$.

a grain-boundary pinning mechanism described by general equations, Eq. (1) and Eq. (2) of the Nb₃Sn layers which will not shift the maximum pinning force towards the higher magnetic flux density [6]:

$$F_p = J_c B = G \frac{(B_{c2}(T))^n}{k^m} f(b) \quad (1)$$

$$f(b) = b^p (1 - p)^q \quad (2)$$

where constant G is a geometrical factor of the microstructure (usually interpreted as a surface area of inclusion per unit volume of matrix), n and m are fitting parameters, k is smoothing parameter defining maximum pinning value, and p and q are pinning and materials related parameters. The position of the maximum of the pinning force on the reduced flux density $b = B/B_{c2}$ is defined by the ratio $p/(p+q)$, and for the particular wire presented in Fig 2: $p = 0.5$ and $q = 2$, which are typical values for grain boundary pinning; B_{c2} is estimated to be 25.5 T at LHe. *Secondly*, there is a central porous core in every filament which contains residual phases and 30% of irregularly distributed voids. (Such voids formation as in all PIT superconductors introduce structurally unsupported core which if exposed to high Lorentz forces is susceptible to degradation of the material and consequently catastrophic reduction of J_c). *Thirdly*, and probably the most decisive factor is that manufacture of the initial NbSn₂ powders for PIT wires is extremely expensive making this process not economic despite very high $J_c(B)$ characteristics in comparison with RRP technology [2].

Grain boundary pinning related mechanism is difficult to change, however there are new possible elaborated approaches that can be further developed [6, 7]. The voids formation in the central core cannot be avoided but can be reduced by implementation of combination of the compositional and architectural changes in the initial filamentary core and also by application of *in situ* external pressure during sintering as it will be discussed in the following sections concerning Bi-based and MgB_2 PIT superconductors. On the other hand, costly manufacture of $NbSn_2$ powders can be overcome more easily with low cost intermetallic manufacture technology. It is obvious that use of the expensive high purity, high melting temperature Nb metal ($\approx 2500^\circ\text{C}$) and low temperature melting temperature Sn (232°C) to produce Nb_2Sn powders presents a challenge. Complex processing and fine powder fabrication methods, makes the overall cost of conventional PIT Nb_3Sn superconductors exceptionally high. A novel electrochemical process was proposed where powder Nb_3Sn superconducting material can be synthesized directly from respective oxide precursors, instead of the high purity metals by means of the solid-state electrode-oxidation method [8]. An example of a simple reduction of Nb_2O_5 to spherical Nb powder is presented in Fig. 3.

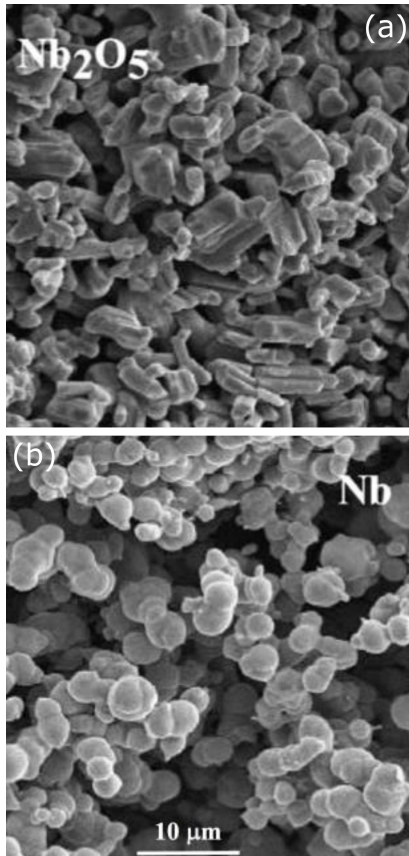


Fig. 3. SEM images of the fractured surfaces representing morphology of the (a) Nb_2O_5 and resulting Nb metal powder, (b) with spherical particles, after electrochemical reduction [9].

This technique is based on the concept of cathodic oxygen ionisation [10]. The new process of making Nb_3Sn conductors is straightforward, less energy intensive, environmentally friendly, and at low operating temperature and low-cost [11]. The same electrochemical reduction can be used to form other superconducting $NbSn_2$ or Nb_6Sn_5 intermetallics from stoichiometric oxide powders to achieve low cost desired $NbSn_2/Nb_6Sn_5$ powder morphology product, making manufacture of the Nb_3Sn PIT conductors economically competitive with half price of current $10 \text{ €}/(\text{kA m})$ at 12 T and 4.2 K [12]. Such price can be lowered further if upscaling of the production takes place but it will increase dramatically if the operating temperature of the superconductor is in range of 14 K.

3. Ag- $Bi_2Sr_2CaCu_2O_{8+d}$ PIT multifilamentary conductor

$Bi_2Sr_2CaCu_2O_{8+d}$ is the only cuprate superconductor that can be made into a round-wire silver matrix conductor with a critical current density J_c over 10^5 A cm^{-2} at 4.2 K. But as it was well documented in literature $Bi_2Sr_2CaCu_2O_{8+d}$ experiences a unique retrograde densification process in the temperature range $850 \div 890^\circ\text{C}$ [13]. Presence of metallic silver interface lowers the binding energy of the Bi-compound accelerating surface diffusion processes as well accelerates formation of thin plate-like single crystallites that grow in a randomly oriented fashion [14] (known as retrograde densification), thus distorting originally precisely manufactured filamentary architecture as presented in Fig. 4. This retrograde densification, coupled with a narrow sintering range [15] and strict oxygen engineering in proximity of the melting temperature [16] makes this compound a difficult one to process.

The resulting microstructure of multifilamentary Ag- $Bi_2Sr_2CaCu_2O_{8+d}$ before sintering looks very regular, see Fig. 4a, whereas after sintering filaments present a very open microstructure with numerous overgrowths bridging filaments and random voids resulting from cuprate migration corresponding to final overall 60–70% density of the superconducting filaments, see Fig. 4b causing significant reduction of $I_c(B, T)$ characteristics.

The only solution to this problem is application of external pressure. There are two methods used: one is by increase the density of the filaments after drawing using 2 GPa of cold isostatic pressure resulting in almost doubling the I_c value, however considering value of the pressure induced before sintering this has no very pronounce effect on the suppression of the retrograde densification [17]. The other more accessible and effective method was to use the hydrostatic gas pressure of $20 \div 100 \text{ atm}$ during sintering up to 887°C that effectively suppressed retrograde densification resulting in 100% dense filaments characterised by dramatically improved I_c almost an order of magnitude [18]. The fact that such a low pressure was sufficient to transfer pressure via metal matrix was that silver melting point is

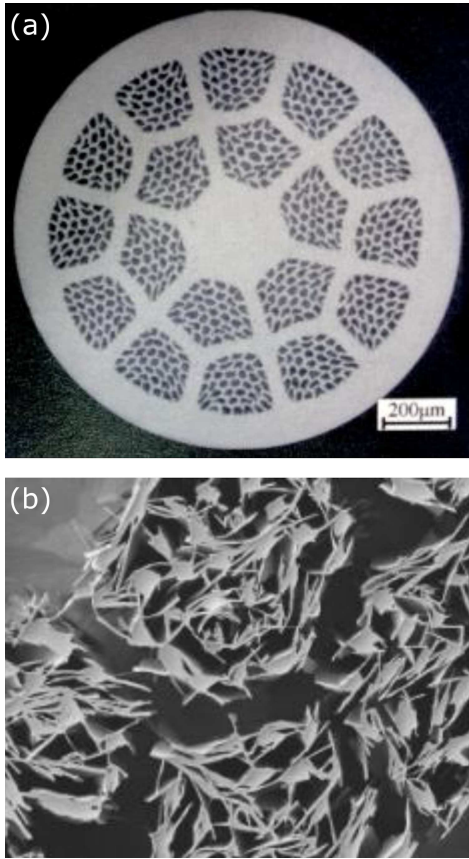


Fig. 4. (a) Image of multifilamentary Ag-Bi₂Sr₂CaCu₂O_{8+x} conductor (18 bundles each containing 37 filaments) in silver matrix before sintering, (b) image of etched silver matrix inter-bundles region after reaction showing result of retrograde densification. Courtesy of L. Motowidlo.

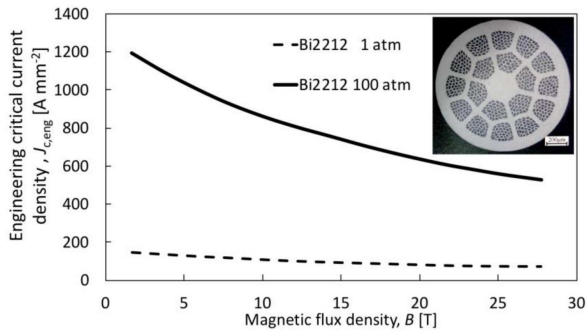


Fig. 5. Improvement of transport engineering critical current density $J_{c,eng}$ value of Ag-Bi₂Sr₂CaCu₂O_{8+d} conductor at 4.2 K, sintered under pressure of 100 atm. Complexity of the retrograde densification of superconducting filaments and their complex morphology makes comparative $J_{c,eng}$ measurements more reliable. (Engineering critical current density is defined as division of I_c over total cross-section of the conductor.) It is evident that $J_{c,eng}$ has increased almost an order of magnitude, outperforming Nb₃Sn PIT conductor above 17 T. The high J_c at high magnetic flux density can only be achieved at LHe temperature whereas the $J_c(B)$ performance at 20 K is comparable to MgB₂. After Larbaestier et al. [18].

≈ 960 °C and the softening of the Ag matrix at sintering temperature of a Ag-Bi₂Sr₂CaCu₂O_{8+x} conductor was only 70 degrees below Ag melting temperature. It becomes evident that combined thermo-mechanical densification process at moderated pressure (in case of soft easy deformable Ag-based matrix) is most effective method to achieve the outstanding densified of round wires for applications at very high magnetic flux densities ≈ 30 T at 4.2 K, Fig. 5.

Of course, one may question the mechanical strength of such silver-based matrix wires exposed to ultra-high Lorentz forces at 30 T, but it is a separate issue, since silver or silver alloys must be used (as an oxygen conductors) to enable optimisation of the oxygen engineering in such wires [16]. Time/temperature dependent pressing procedure following Ashby deformation maps has been also used to successfully increase the density of bulk Bi-2223 samples [19], that suffer less pronounced but similar retrograde densification as Bi₂Sr₂CaCu₂O_{8+d}. This was an inspiration for development of overpressure densification of multifilaments in Ag-Bi2223 tapes. In this process application of controlled overpressure sintering (CT-OP) (at pressure of 300 atm and 900 °C) enables achieving a very dense, well-interconnected superconducting core in the individual filaments [20]. Such a procedure enhanced the critical current by 50% if compared to conventional Bi-2223 wires sintered in atmospheric pressure, and most importantly that voids disappeared almost completely in the CT-OP processed specimens. To conclude, the detrimental effect of retrograde densification of round PIT Ag-Bi₂Sr₂CaCu₂O_{8+d} conductors can be effectively overcome by application of *in situ* thermo-mechanical treatment. The only problem is the price of such wires because: approximately 1/3 of the cost is materials, 1/3 is an intensive labour and 1/3 electricity which cannot be altered and the price at 4.2 K and 0 T is estimated to be ≈ 40 €/kA m [21]. This price will increase significantly if conductor operates at 14÷20 K range. Therefore, other PIT conductors that can deliver a similar performance at 20 K at substantially lower price can become superconductors of choice for hydrogen-cryomagnetic technologies or even cryogen-free technologies.

4. MgB₂ *in situ* PIT densification methods

PIT Nb₃Sn and MgB₂ superconducting materials are not very different if one considers that both of them are made via *in situ* processes that required reactive solid-liquid diffusion of elements and compounds with involved phase transformation. In the case of MgB₂ it is simply the matter of Mg diffusion to B with assistance of additives such as SiC, C, and Cu nanoparticles to improve magnetic flux pinning at higher magnetic flux densities, whereas in case of Nb₃Sn it is a solid-liquid reactive diffusion of Sn atoms from NbSn₂ in presence of Cu acting as a catalyst and fine grain boundary facilitator [5] to form with Nb a Nb₃Sn layer. The importance of technological comparison between PIT Nb₃Sn and PIT MgB₂ liquid Mg reactive infiltration process (Mg-RLI) [22] seems to

be more relevant that comparison of MgB₂ that has a tag price of > 5 €/kA m) with NbTi that have tag price of 1 €/kA m).

In this section, we would conduct an analysis of methods used to improve properties of the *in situ* MgB₂ conductors. MgB₂ wires manufactured by the *in situ* technique, in contrast to *ex situ* wires, diffusing magnesium atoms to boron particles experienced ≈ 25.5% decrease in density from the initial value after cold deformation, due to the phase transformation from Mg + 2(β-B) → MgB₂ [23]. In case of the typical PIT process where particles of Mg are few tenths to hundred micrometre sizes, the formation of the resulting voids in the volume of the conductor create percolated network, Fig. 6a, weakening the electromagnetic performance of the conductors.

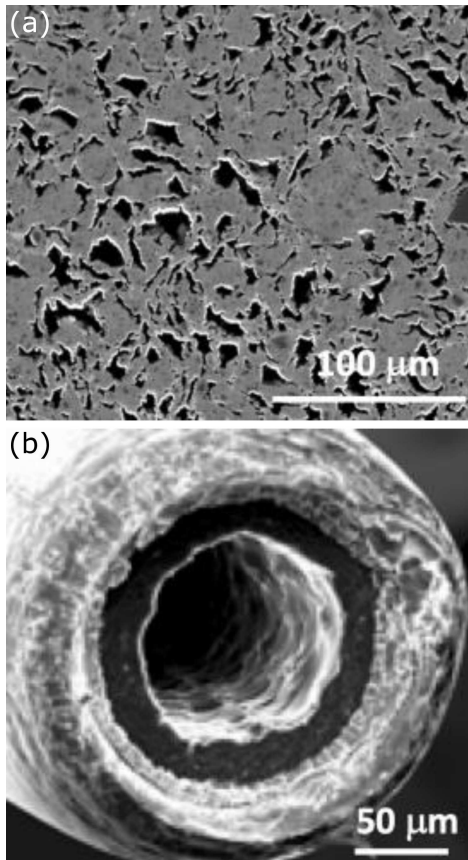


Fig. 6. SEM image of (a) the central core *ex situ* Monel/Ti/MgB₂ PIT wire [24] (the black areas represent porosity), (b) fractured cross-section of *in situ* Mg-RLI MgB₂ wire showing central void after Mg diffused to the boron powder, the black ring represents dense MgB₂ layer adopted (courtesy of G. Giunchi).

To eliminate the distributed voids created in *in situ* MgB₂ PIT conductor a Mg-RLI process was used, where actual MgB₂ monolithic ring can be created with a central single residual void instead, see Fig. 6b [22]. Critical current of such a conductor reaches the highest value, due to elimination of percolated path and fine grain

structure similar to PIT Nb₃Sn conductors. However, problem with unsupported central hole and rather low J_{eng} remains. The *ex situ* wires do not suffer from such a retrograde transformation but densification and consolidation of the MgB₂ powder in PIT technique in general require compaction and in most cases subsequent sintering at elevated temperatures of 800 °C and above which narrowing down options of the materials used for the composite PIT process. However, it was well documented in literature that cold deformation of MgB₂ powders in PIT wires without any sintering enabled remarkable transport critical current density value of MgB₂ to reach 1×10^3 A mm⁻² at 4.2 K [25]. But sintering of the *ex situ* bulks shows “solid-state self-sintering” of the MgB₂ grains with dispersion of porosity in the grains, but without grain growth reaching $J_c = 5 \times 10^3$ A mm⁻² at 20 K [26]. Such findings present an opportunity to explore the possibility of combined *ex situ* and *in situ* technologies to thermo-mechanically fuse together [(MgB₂)]/[(Mg+2B)] core in PIT copper sheathed wire.

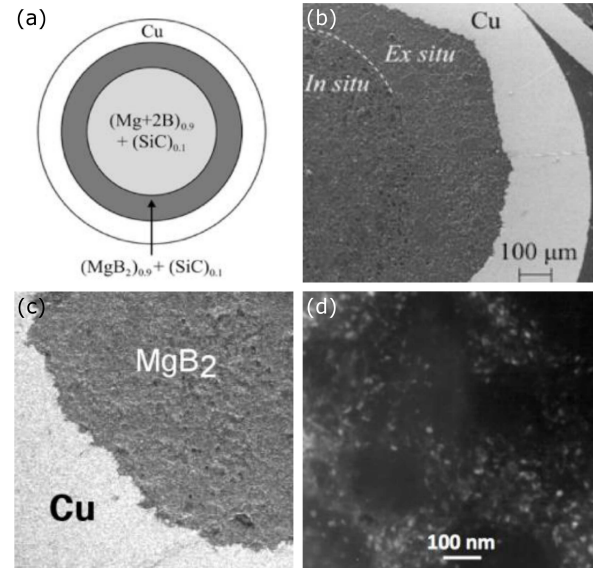


Fig. 7. Images of Cu/[(MgB₂)_{0.9}(SiC)_{0.1}]/[(Mg+2B)_{0.9}(SiC)_{0.1}] composite wire with SiC 18 nm average grain size powder: (a) schematic of the overall cross-section, (b) SEM micrograph of part of the cross-section, showing the distinctive difference in morphology between *ex situ* and *in situ* cores before reaction [28], (c) SEM micrograph of MgB₂ core after reaction, (d) TEM micrograph showing MgO nano-inclusions population in the *ex situ* region, indicating one of the source of artificial pinning centres embedded into the MgB₂ microstructure as observed by Komori et al. [29].

As none of the above described MgB₂ preparation process can avoid formation of voids (as a result of phase transformation from Mg + 2(β-B) → MgB₂) a novel approach was adopted, similar as described in previous section, and used to hydrostatically densify Ag-Bi₂Sr₂CaCu₂O_{8+x} during sintering that results in 100% dense filaments characterised by dramatically im-

proved I_c almost order of magnitude. In MgB_2 case metal matrix should prevent oxygen diffusion in contrast to $\text{Ag-Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+x}$ but to achieve a similar effect in terms of sheath metal softness at elevated temperatures, Cu matrix was chosen. Additionally, to prevent formation of the unwanted MgCu_2 phase between Cu and Mg [19] and also eliminate need for diffusion barrier, a patented concept of the composite $\text{Cu}/[(\text{MgB}_2)_{0.9}(\text{SiC})_{0.1}]/[(\text{Mg}+2\text{B})_{0.9}(\text{SiC})_{0.1}]$ conductor was used in which a central *in situ* $\text{Mg}+2\text{B}$ core was surrounded by a concentric *ex situ* MgB_2 tube acting as a diffusion barrier, Fig. 7 [27], see Fig. 7a and b.

Starting *in situ* powder was densified using cold isostatic pressing at pressure of 0.3 GPa assuring better density of the central insert and higher uniformity of the external *ex situ* MgB_2 layer. A dynamic sintering was conducted at 750 °C for 15 min under pressure of 1 GPa. From an earlier experiment, it became evident that pressure needs to be introduced before *in situ* formation of the MgB_2 is taking place and preferentially continues during high temperature sintering to result in densified core [30].

It can be argued that in the configuration where parts of *ex situ* and *in situ* Mg-B conductor coexist, process of MgB_2 formation and co-densification of MgB_2 under high pressure and short time can be very complex to achieve the desired high I_c value across the [*ex situ/in situ*] HIP conductor, Fig. 7. Such process requires highly dynamic non-isothermal rapid sintering to satisfy infiltration of the *ex situ* part by some magnesium atoms as well as induce pressure on the *in situ* core via *ex situ* layer. Such sintering process must allow formation of the MgB_2 and Mg-assisted densification of the whole conductor under induced gas pressure.

Resulting microstructure of MgB_2 *ex situ/in situ* PIT wire, Fig. 7c is not clearly understood, however, disturbed crystal structure, chemical deviation, the presence of distributed secondary non-superconducting phases, the presence of stacking faults and numerous structurally induced macro-voids as well as nano-sized MgO (see Fig. 7d) were also observed in thin films [29]. (Anisotropy of the electronic properties at high-angle boundaries of the crystals can all contribute to high values of critical current of the resulting MgB_2 core as a whole, Fig. 8.) Volume flux pinning computer simulation has revealed co-existence of two pinning mechanisms: grain boundary and also APC in analogy to pinning mechanisms in thin films [25, 29, 31] shifting the maximum pinning force towards the higher magnetic flux density.

4. Conclusions

It becomes apparent that analysed three powder-in-tube superconductive conductors can potentially be competitive at 14–20 K if novel effective thermally-assisted densification processes can be used. MgB_2 at 4.2 K can possibly even compete with other discussed PIT conductors in the high magnetic flux density range if novel on-line densification process is developed. Hot pressing

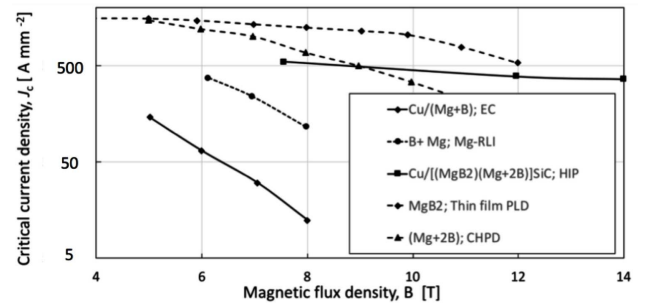


Fig. 8. Comparison of the transport current performance results of the Cu based wires technology (solid lines) with other PIT and thin film technologies (dotted lines), vs. magnetic flux density at 4.2 K. The PIT $\text{Cu}/[(\text{MgB}_2)/\text{Mg}+2\text{B}]$ hot isostatically pressed (HIP) wire performance at higher magnetic flux densities is reaching J_c of MgB_2 manufactured by cold high pressure densification (CHPD) [32], approaching values for pulse laser deposited (PLD) thin films [29] and also outperforming reactive liquid Mg infiltration (Mg-RLI) wires [22] as well as electromagnetically compressed (EC) copper clad wires [31]. In this case due to complexity of comparison of the very different conductor architecture, substrate and matrix relative volume as well as MgB_2 density variation, the values of the critical current density presented correspond to the actual part occupied by MgB_2 superconductor..

increases density by increasing the stresses present at the particle contacts, adding plastic yielding and power-law creep to the pressure-less diffusional densification mechanisms.

Intensive research and development of all three conductors should continue not only for the benefit of the potential applications especially in the range of 14–20 K in anticipation of the emerging hydrogen cryo-magnetics revolution for energy applications, but also for better understanding of influence of novel reactive thermo-mechanically assisted densification processes on microstructural and electromagnetic properties of superconducting materials. However if advanced MgB_2 wires can deliver desired current density at 15–20 K at moderate and higher magnetic flux densities, at target cost as low as 2 €/kA m), then MgB_2 will emerge as replacement PIT technology for applications benefiting from elevated temperature application margin, relying on cryogen-free, close loop helium gas cooling or even liquid hydrogen cooling.

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