STATUS AND PERSPECTIVES FOR TECHNICAL USE OF MgB_2 WIRES

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Abstract
Since the announcement of the discovery of superconductivity in MgB_2 in 2001 a lot of work has been done worldwide to develop wires and tapes for technical applications. The comparatively high T_c of 39 K and the absence of weak link behavior in MgB_2 raise hope to use MgB_2 as a cheap conductor at elevated temperatures around 20 K, replacing or complementing NbTi wires or HTS tapes. Although first applications of MgB_2 wires are currently arising, many obstacles have still to be overcome before MgB_2 can be used as a technical conductor. The main efforts worldwide concentrate on the improvement of current carrying capability, enhancement of irreversibility and upper critical field and on the improvement of thermal and mechanical stabilization of the conductors.
In this paper we illustrate the current status of MgB_2 wire and tape development and present a first technical application. As an outlook we highlight the perspectives for technical MgB_2 wires and for using these conductors in magnets, current leads, etc.

1. INTRODUCTION

The discovery of superconductivity in the binary compound MgB_2 by Akimitsu et al. 2001 [1] initiated a worldwide intensive investigation of this material as thin film, bulk and from the beginning also already as a wire or tape composite. The critical temperature of 39 K, the highest among many superconducting borides and borocarbides so far [2], motivated the R&D activities due to the chance having a new superconductor in between the LTS and HTS materials which can be cooled cryogen free with GM- or pulse-tube coolers to an operation temperature of about 20 K. The hope to raise the critical temperature by substitutional alloying was not successful yet and will be limited to negligible effects regarding the state of the art knowledge about the properties of MgB_2. Although MgB_2 has an anisotropic layered hexagonal crystal structure (P6/mmm) with alternating Mg and B layers (see Fig. 1)

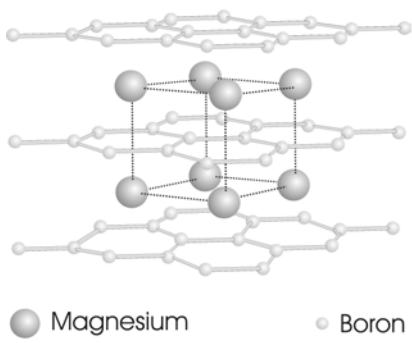

Fig. 1: AlB_2-type crystal structure of MgB_2 with alternating Mg and B layers

and anisotropic properties with respect to thermal expansion, compressibility, resistivity, critical current, irreversibility field and upper critical field, granularity as in the HTS perovskite $YBa_2Cu_3O_x$ was not found. The explanation for this positive feature is a sufficient large coherence length of $\xi_{ab} = 10$ nm (in plane) and $\xi_c = 5$ nm (out of plane parallel *c*-axis) [3]. Therefore already quite early high transport currents were realised in wire and tape composites made by the powder in tube (PIT) technique applying different sheaths of Fe, Nb, Ta, Ni, Monel, Cu, steel, etc. and precursors from commercial MgB_2 (ex-situ method) and Mg+B powder mixtures (in-situ methods). The magnetically measured current densities in self field exceeded 10^6 Acm^{-2} at 4.2 K, which is comparable to the technical and commercial low T_c superconductor NbTi. For MgB_2 conductors a heat treatment of the final wire at $T = 600$-$900°C$ is applied to sinter the MgB_2 grains to a compact filament (ex-situ route) or to react the phase from the elements regarding the in-situ route.

Thin films, commonly *c*-axis textured, gave the first indication for quite high upper critical fields of 40 T ($H \parallel a,b$) [4] and more in this material. The irreversibility field in thin films, wires and tapes is usually 5-10% lower than H_{c2}. Recent theoretical considerations on the paramagnetic limit expect possible upper critical fields up to 70 T [5]. In a specific thin film sample the critical current density of 200 $kAcm^{-2}$ (self field) showed a surprising low field dependence up to high fields of 10-12 T at 4.2 K, indicating that effective flux pinning is a key to improved conductors and a sufficient thermal stabilisation necessary at low fields to avoid thermal driven transitions. The results in thin films however still scatter in a large range, since the applied different preparation methods are not trivial and not reproducible due to the difficulties in processing the quite volatile element Mg in a vacuum deposition process.

In the first wires and tapes upper critical fields were only in the range of 15 – 17 T [6]. The quite recent progress of the preparation methods towards small sized MgB_2 grains led to significantly improved upper critical fields and irreversibility fields.

For technical conductors, additional properties as mechanical strength and thermal stabilization are important. The mechanical properties depend strongly on the composite structure, the choice of the sheath materials and the heat treatment temperature. The critical currents show a slight reversible degradation under the compressive prestress originating from the sheath [7]. A moderate steel reinforcement can enhance the mechanical properties of the wires significantly, higher steel contents lead to irreversible degradations of the critical current density [8]. An effective or sufficient thermal stabilization of MgB_2 wires is presently not given and would require a multifilamentary structure with very thin filaments of a few micrometer in diameter and a low resistance sheath which can carry the normal current above I_c. These properties are not realized yet, for high transport currents in low fields most conductors quench well below the critical current and burn through in the high current regime (low temperature and field).

2. PREPARATION METHODS FOR MgB_2 CONDUCTORS

MgB_2 conductors are prepared by means of the powder in tube technique (PIT), with the choice of two different precursor routes, either with pre-reacted MgB_2 powder (ex situ method) or with powder mixtures of Mg and B (in situ method). The powder is filled into a metal tube and deformed to a wire or tape. Ex-situ or in-situ refers to the phase formation before manufacturing the PIT conductor or forming the phase in the final conductor from the elements applying a heat treatment, respectively. For some improved preparation routes Mg alloys [9] or boron compounds [10] are used as precursors or the precursors are mixed with fine powders of a secondary minority phase [11].

The ex-situ method has the advantage that the MgB_2 particle size can be adjusted by grinding processes, leading to small grains in the filament for enhanced flux pinning [12]. With this route cold worked ex-situ conductors can be produced, which are not only suitable for a cheap industrial production process but also allow the use of a wide selection of sheath materials [13]. However, cold worked PIT conductors have a limited filament densification, much lower currents and poorer

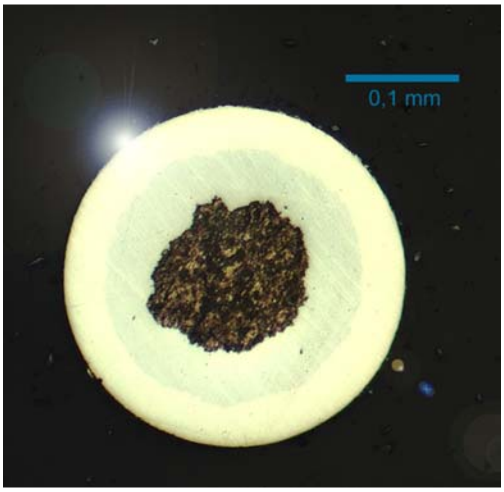

Fig. 2: Optical micrograph of a MgB_2/Fe/SS current lead wire cross section (Ø = 310 µm)

mechanical properties. Therefore the application of a heat treatment is commonly preferred since it provides well connected grains [14] and better mechanical properties of the conductor [15]. The ex-situ route has the disadvantage that favourably a heat treatment has to be applied at quite high temperatures of 900-950°C to sinter the MgB_2 grains dense via a decomposition-reformation process. However, since MgB_2 has no defined melting point and decomposes at T > 900°C the sinter effect is limited and the danger of thick chemical reactions with the sheath is favoured, as commonly observed.

The in-situ phase formation takes place at much lower temperatures of 600-650°C and has the advantage that the in-situ MgB_2 formation leads automatically to very well connected grains. A disadvantage of this route is that the final MgB_2 grain size in the filament depends on nucleation and grain growth and the mixture and grain size of the Mg and B powders. Actually the in-situ method is more and more preferred since the chemical reaction with the sheath is reduced for the low-temperature processing methods and a good grain connection is achieved [16].

Both fundamental conductor geometries, round wires and flat tapes are developed by the different

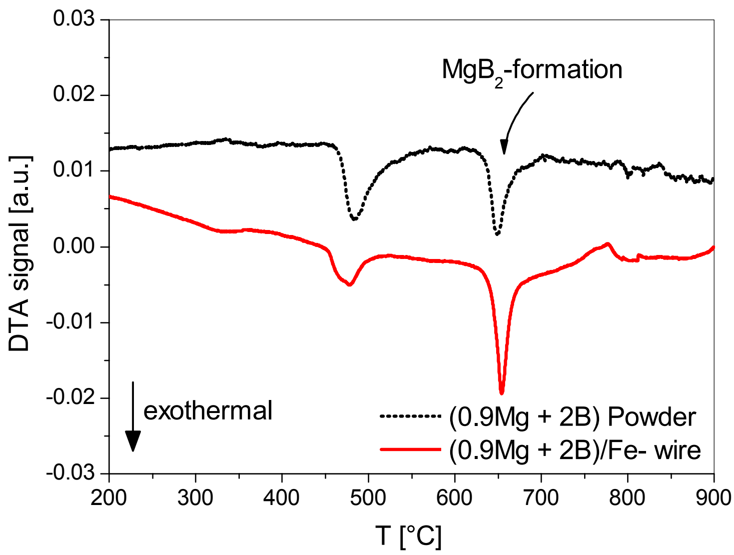

Fig. 3: DTA characterisation of a MgB_2/Fe wire and the corresponding Mg + B precursor powder mixture

Fig. 4: SEM image of MgB_2 phase in the filament of a MgB_2/Fe wire

groups worldwide. Tapes give the chance to take advantage of the anisotropy of the superconductor via a textured MgB_2 phase [17]. Although indeed a current anisotropy was reported in tapes, especially in higher fields, the advantage for the critical currents is limited, since the mechanism achieving the texture is a mechanical orientation of grains close to the filament sheath interface and the texture effect is limited to this part of the filament. However for future much thinner filaments this option has to be considered again. Another advantage of tapes is the high densification of the filament material due to the high rolling pressure during deformation which improves the density of MgB_2 [18]. Therefore tapes often show higher current densities compared to wires. Round wires are the preferred conductor geometry for layered windings in coils and much better suited for low AC loss conductors, especially for applying a filament twist.

As sheath materials Fe, Cu, Nb, Ta, Steel, Ni and different alloys are used, which allows or requires different annealing temperatures. Fe has the overall best properties and Cu a stronger chemical reaction with the filament during heat treatments, Nb and Ta negligible chemical reaction but an unfavourable thermal expansion coefficient and Steel and Ni in contact to the filament react strongly during the heat treatment and are used for cold processed conductors only.

Multifilamentary conductors were rarely presented so far since the current densities are commonly smaller compared to monocores due to filament inhomogeneities and irregularities of the geometry from sausaging effects.

3. METHODS TO OPTIMISE THE CRITICAL CURRENT DENSITIES

As in the classical LTS superconductor Nb_3Sn, flux pinning in MgB_2 occurs at the grain boundaries or defect structures and scales with the grain size or defect density. Different methods can be applied to improve flux pinning. Successful examples are:

- Controlled phase formation: a control of the nucleation and growth process of MgB_2 in in-situ processed wires from commercial powders was achieved by applying a heat treatment with partial phase reaction during the deformation and a final heat treatment at quite low temperatures of 640°C, the low temperature onset of the phase formation regime, which favours nucleation and limits the grain growth. For round wires with commercial Mg and B precursors the highest current densities were achieved comparable to the best tapes.

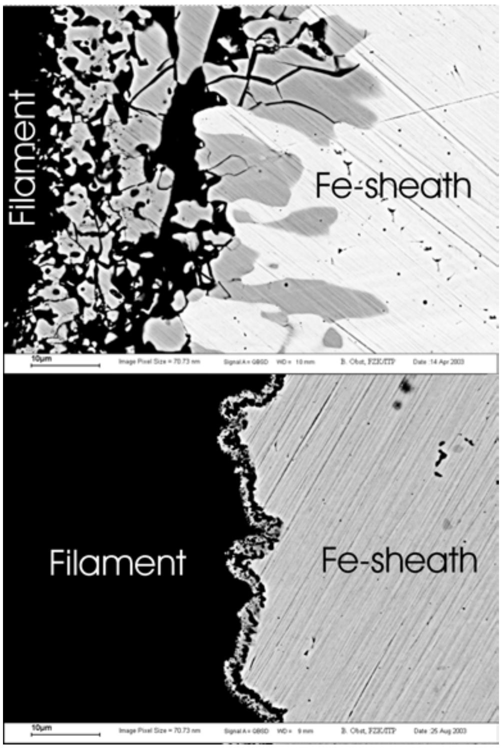

Fig. 5: Reaction layer at the filament sheath interface of MgB_2 /Fe wires heat treated at 905°C (top) and at 640°C (bottom)

- Neutron irradiation of such wires created an additional defect structure in the scale of several nanometers and led to significantly further improved current densities, irreversibility fields and upper critical fields, indicating that the density of flux pinning centers was not at an optimum in the unirradiated wires [19, 20].
- Mechanically alloyed precursors (intensive milling of powders from the elements) led to a mixture of the elements in the nanometer scale and a beginning phase formation. DTA measurement showed that the reaction temperature was significantly reduced to about 500°C [21].
- SiC secondary phase particles in the nanometer scale added to the filament in a fraction of 5-20% is presently the most effective method to create homogeneously distributed pinning sites and enhances the current densities by nearly one order of magnitude at higher fields [11, 22].
- Improved Mg precursor powders processed by plasma spraying with a grain size of 300 nm, induced also small MgB_2 grains and improved the current densities and the critical field values H_{irr} and H_{c2} [22].

For some examples and routes also a decrease of the phase formation temperature was observed. An example is the tape from mechanically alloyed powder [21].

There are additional more or less effective combinations of MgB_2 with secondary phase particles which proof that particle additions in the nanometer scale, which do not react with MgB_2 during the heat treatment, are a practical way to limit the grain size of the MgB_2 phase and to enhance the density of pinning sites in the filament.

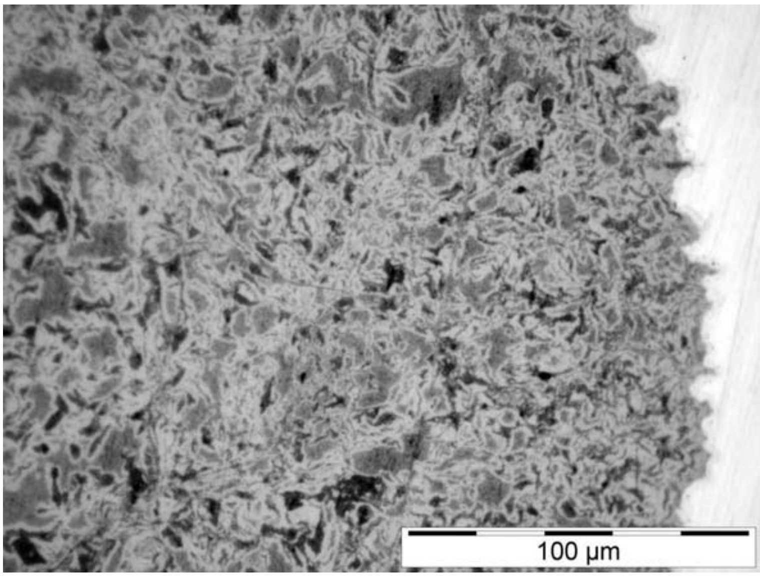

Fig. 6: Optical micrograph of the filament section of a MgB_2/Fe wire heat treated at 640°C showing the meander like layered MgB_2 phase (light colour) and the Boron rich secondary phase (dark grey colour)

4. EXAMPLE OF A MECHANICALLY REINFORCED IN-SITU MgB_2 WIRE WITH THIN FILAMENTS FOR SPACE APPLICATION

In this section we present some characteristics and limitations of MgB_2 conductors taking as an example a very thin stainless steel reinforced MgB_2 wire, developed for a satellite application. In this case a mechanical reinforcement and a wire thickness as small as possible to keep the thermal conductance low and an operation current of 2 Amps DC below 17 K was demanded.

4.1 In-situ preparation applying a low-temperature heat treatment

Thoroughly mixed commercial Mg powder and amorphous Boron powder (both -325 mesh) with an atomic Mg/B ratio of 0.9/2 were filled in Fe tubes of 99.5% purity (10 mm outer diameter, 1.5 mm wall thickness) and swaged down to 1.8 mm with two intermediate heat treatments at 600-620°C to release stresses in the Fe sheath. During these heat treatments a partial reaction of the MgB_2 phase takes place. In a next step the wires were inserted into precision stainless steel (SS) tubes (AISI 316) of 2.5 mm outer diameter and 0.3 mm wall thickness and drawn to a final diameter of 310 µm (see Fig. 2 with a further intermediate heat treatment to release stresses in the sheath. The final low temperature annealing was performed at 640°C for 1h with subsequent furnace cooling. To limit the reaction kinetics, the annealing temperature was set at the low temperature onset of the exothermal phase formation dip in the DTA curve which has its minimum at 650°C, corresponding to the melting point of Mg, as shown in Fig. 3. The final wire cross section is composed of approximately 20% MgB_2, 40% Fe, 40% SS, the filament diameter is approximately 140 µm. During deformation samples were extracted from the batch at some thicker wire diameters and annealed to investigate the influence of the filament thickness on the current carrying capacity. SEM pictures of the filament microstructure showed MgB_2 grains in the scale of 10-20 microns, an indication of an effective nucleation (Fig. 4). The reaction with the Fe sheath was strongly reduced to a layer of only 1-2 µm thickness (20-30 µm for annealing at 900°C) as shown in Fig. 5. The resulting microstructure of MgB_2 looks layered like a pastry, obviously forming an effective percolation path for the supercurrents (Fig. 6).

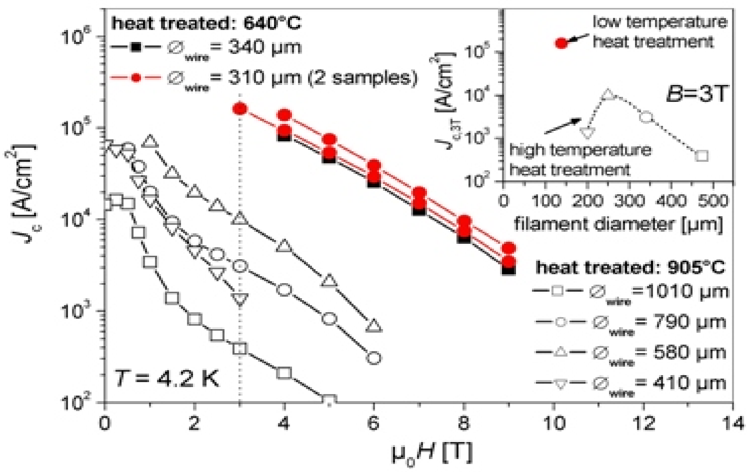

Fig. 7: Transport critical current density $J_c(B)$ of current lead wires, old batch = high temperature heat treatment at 905°C (3 samples having thicker filaments for comparison) and new batch = low temperature heat treatment at 640°C. The insert shows the correlation of the critical current density with filament diameter at B=3 T

4.2 Enhanced transport critical currents, irreversibility fields and upper critical fields

In Fig. 7 the transport critical current densities of in-situ wires with small filaments (∅ = 310 µm and 340 µm) and low temperature heat treatment and the former batch (high temperature annealing) from ref. [16] are shown together. In the insert the dependence of the current densities on filament thickness is given. For the former batch the critical current density increases with decreasing diameter down to 200-250 µm, but further reduction of the filament diameter leads to a decrease of J_c. An

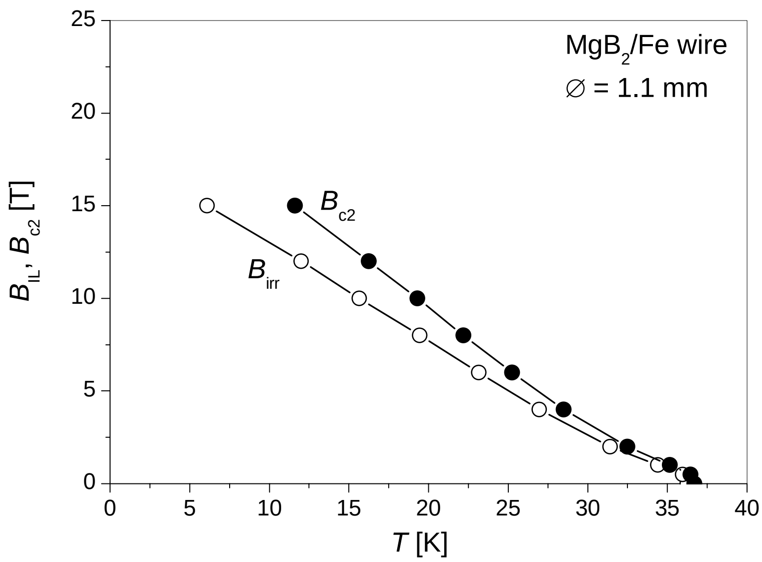

Fig. 8: Irreversibility field B_{irr} and upper critical field B_{c2} with temperature obtained from electrical resistivity transitions for a MgB_2/Fe wire. The values were determined as described in the text

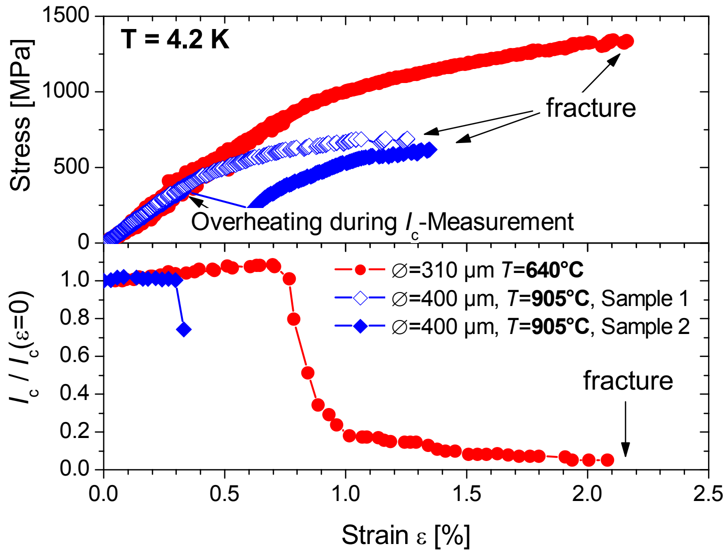

Fig. 9: Critical current and tensile stress vs. axial strain for an MgB_2 current lead annealed at low temperatures ($T = 640°C$). Equivalent data for 2 samples annealed at high temperatures from ref. [16] are added for comparison

critical current densities were 150 kAcm^{-2} at 4T, 40 kAcm^{-2} at 6 T and 10 kAcm^{-2} at 8 T (4.2 K) for the 310 µm thick wire. However below 3-4 T the wires are not sufficiently stabilized for high transport currents and burn through exceeding a definite current level.

The field dependent resistive T_c transitions $R(T,B)$ of the wires with 650-750 µm thick filaments (wire diameter 1.1 mm) were measured with small constant DC currents of $I = 30$ mA. For each magnetic field the temperature for the upper critical field $T(B_{c2})$ was determined from $R(T)$ as the temperature where $R = 0.9 \cdot R_{normal}$ and the temperature for the irreversibility field $T(B_{irr})$ was determined as the temperature, at which the electric field between the voltage tabs is 1 µV/cm. In Fig. 8 these data are plotted as $B_{c2}(T)$ and $B_{irr}(T)$. A linear extrapolation of $B_{c2}(T)$ and $B_{irr}(T)$ to $T = 0$ K gave values of 18 T and 22 T respectively, which are remarkable high for non doped MgB_2 round wires made from commercial non modified powders.

4.3 Mechanical properties

The critical current as a function of axially applied strain given in Fig. 9 was measured at 4.2 K in small background fields of 0.5 or 1 T (to reduce the currents and to avoid overheating). Measurements of two wires from the former batch with different steel contents (40 % and 30 %) are also shown. The former samples showed already a degradation of the critical current at 0.27 % and 0.3 % strain since the steel clad was softened through the high annealing temperature. With the first strain induced crack in the filament one sample burned through immediately and the second became hot in the crack section, which caused plastic deformation in the sheath and a related drop in the load display. The second wire with already broken filament could be strained further until final fracture at 1.5 % strain. The tolerable stresses were 320 MPa and 350 MPa, respectively, and the yield strength (0.2 % strain) was 220 MPa.

Contrary the wire with low temperature treatment showed a strongly improved mechanical performance. Tolerable strains of 0.7 % without current degradation were observed, corresponding to stresses of about 800 MPa. The main reason for this improvement is attributed to the low annealing temperature of 640°C, which is low enough to avoid softening of the stainless steel clad. The crack

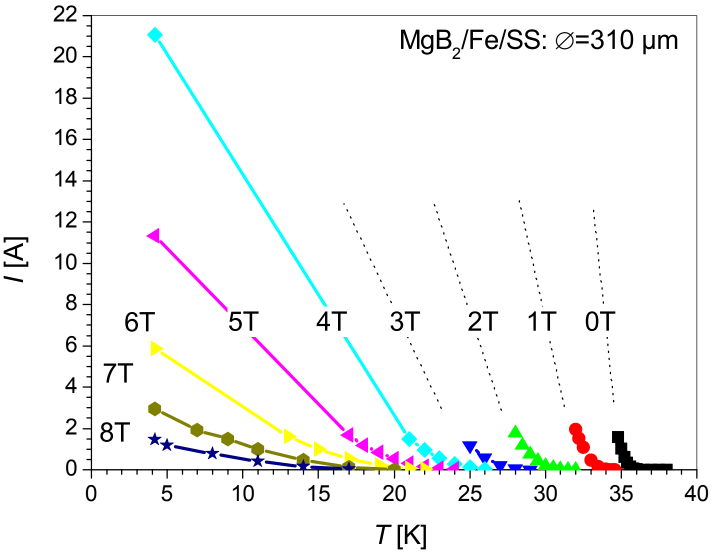

Fig. 10: Transport critical currents of MgB_2/Fe/SS current lead wires with field and temperatures. At $T > 4.2$ K the currents were measured with a Quantum Design PPMS device (I limited to I_{max}=2A)

formation in the filament occurs steadily and not as abrupt as it is the case in the former wires, obviously favoured by an effective transverse compressive stress component in the filament which keeps the filament material compact.

The recovery of the critical current density upon strain can also be understood. It was shown that T_c is reduced under pressure in particular under non hydrostatic stress conditions. [24,25]. If the precompression of the thermal stress of the matrix is compensated by applied external tensile stress, improved superconducting properties are the consequence. This behavior is very similar to Nb_3Sn wires or Chevrel phase wires and very favourable for technical use.

4.4 Thermal stability

A detailed investigation of the whole $E(I)$ transition in MgB_2 conductors gave some information about the onset and contribution of thermal effects. Such effects were found at all current levels and background fields and depend on the transport current density level [26]. At high transport currents, thermal effects can significantly increase the slope of $E(I)$. As a consequence the calculated n-values become larger and may be overestimated. A contribution of such a successive energy dissipation to the shape of the $E(I)$ transition is very difficult to detect especially for small E-values, the observed heat fluctuations however indicate their presence. The comparison of different MgB_2 monofilamentary wires showed that both a homogeneous microstructure and a high current carrying percolation path have a significant influence on the intra-filamentary thermal stabilisation, the occurrence of hot spots and the dissipation of the energy approaching I_c. The distribution of the local critical currents and current density, a consequence of the microstructure, especially inclusions of secondary phases, plays an important role for the ignition of the complete quench at high transport currents. For the low temperature heat treatment surprising low n-values of only 15 not depending on background field were observed in one sample batch. An explanation might be the presence of an effective percolation path and an intra-filamentary current sharing at I_c [26].

For the presented thin MgB_2 wire, transport critical currents at higher temperatures and in fields are given in Fig. 10 and n-values determined from the I-V curves are shown in Fig. 11. Plotting the n-values versus transport currents indicates the presence of thermal effects by the deviation from a linear behaviour (left side of Fig. 11). The large reserve for the demanded transport currents of 2 Amps at 17 K and below (seeFig. 10), allows the use of these wires in the satellite application.

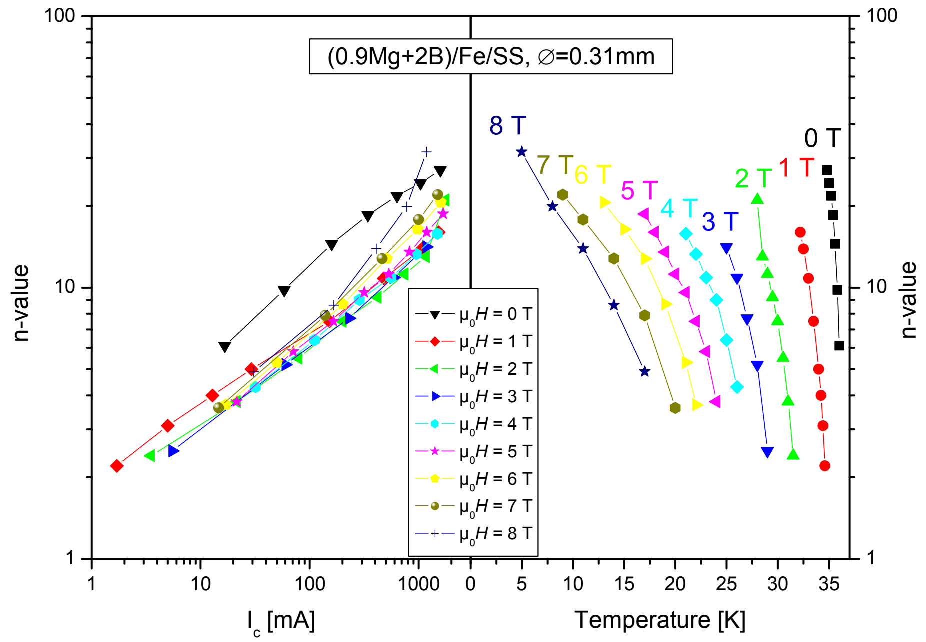

Fig. 11: *N*-values calculated from *E(I)* transitions in the range 0.5-5 μV/cm for the critical current data of Fig.10, plotted versus transport critical current (left side) and temperature (right side)

For a final statement about the true intrinsic *n*-values of MgB_2 filaments and for the development of a stabilised technical conductor, a multifilamentary structure with thin filaments, homogeneous microstructure, regular filament geometry and a sufficient external thermal stabilisation is absolutely necessary to eliminate the contribution of heat generation to the I_c transition in the filaments.

5. *COMPARISON WITH THE MOSTLY ADVANCED FABRICATION METHODS*

As already mentioned above, different methods were successful to enhance the critical current density in MgB_2 wires and tapes. Already an optimisation of the precursors using smaller particles [22, 12] or applying low temperature heat treatments [16] leads to smaller grains in MgB_2 and an enhancement of the critical current densities and the critical fields, irreversibility field and upper critical field. Neutron irradiation induced in addition a defect structure in the scale of a few nanometer, which led to a further enhancement of the critical current density and shows that the flux pinning site density was still too low. More effective is doping of MgB_2, in particular with SiC particles of nanometer size. Comparing the critical current densities of the advanced MgB_2 conductors in Fig. 12 illustrates the effect of improved pinning, the slope of the *I*(*B*) curve becomes smaller which correlates with higher current densities at higher fields and improved H_{c2}. However, all advanced conductors have a common disadvantage, the onset of the critical temperature is shifted to lower values by up to a few Kelvins. This effect has negative influence on the critical currents and upper critical fields at 20 K. Table 1 gives some characteristic data for the advanced conductors.

Table 1: Comparison of the critical current densities $J_c(B)$ of MgB_2 /Fe wires and tapes with different precursors at 4.2 K and 20 K.

Method	**J_c [kA/cm^2] at 4.2 K, 7T**	**J_c [kA/cm^2] at 4.2 K, 9T**	**J_c [kA/cm^2] at 20 K, 3T**	**Reference**
Commercial Mg, commercial B	20.6	5.8		This Work
Mechanical alloying	39.7	11.6	9.4	Fischer *et al.* [21]
10 % SiC-doping	58.5	16.2	60	Dou *et al.* [11]
Nano-Mg Commercial B	48.9	27.6		Yamada *et al.* [22]
Nano-Mg + 10 % SiC-doping		40		Yamada *et al.* [22]

6. *OUTLOOK TOWARDS TECHNICAL WIRES AND APPLICATIONS*

With the example of a thin reinforced MgB_2 wire for a satellite application, the possibly first application of MgB_2 was achieved. The mechanical data were excellent meeting all technical requirements so far. In this special application a large reserve of the transport current was given, which allows the risk to use a non thermal stabilized conductor.

One of the main challenges to approach technical MgB_2 conductors which possess all necessary features is an improved preparation technique for a multifilamentary composite structure with small filaments and regular filament and conductor cross section geometry. Sausaging of the filaments, a typical hint of the PIT route, and secondary phase inclusions have to be avoided since both serve as a source for hot spots and thermal instability. So far multifilamentary conductors are limited to 7 or 19 filaments and show a degradation of the critical currents compared to the monocores. In addition a low resistive sheath, preferably Cu, is required in contact with the filament to enhance the thermal stabilization. Reaction layers between filament and sheath have to be eliminated to support the current sharing and energy transfer at the I_c transition. As a consequence improved low temperature preparation routes are required to restrict or avoid the chemical reaction. Modified precursors already showed some progress in this sense and provide an optimistic outlook for improved conductor preparation routes in the near future. Advanced precursors are also asked for an improvement of the MgB_2 microstructure and the incorporation of pinning sites in the nano scale. Further increased values for H_{irr} and H_{c2} in wires approaching the dirty limit and the values already measured in thin films, are crucial for a breakthrough of this material in competition to LTC conductors.

Since the improvement of the current density and critical fields through small MgB_2 grains or doping correlates with a decrease of the critical temperature by a few degrees the application at higher temperatures as 20 K cannot take full advantage of the improvement of the transport current and critical fields. Transport currents and critical fields scale almost linear with temperature between 4.2 K and T_c. From this situation one can estimate that actually an application at 20 K is limited to background fields of about 5 Tesla if the best conductor performance is available in long lengths.

Due to the absence of granularity in MgB_2 the realisation of persistent mode contacts connecting conductor ends seems possible. This is the motivation for the interest in one of the most promising future applications, persistent mode low field MRI coils for 20 K operation. The demonstration of a small MgB_2 coil, which created a field of 1 T at 4.2 K was presented recently [27].Another promising application in energy technique might be the fault current limiter, also operated at temperatures around 20 K. MgB_2 wires are quite suitable for this purpose since the resistivity of the sheath can be selected and adjusted via the choice and combination of the material. If thermally stabilised wires are available the application in motors, generators, transformers and short cables will surely also be

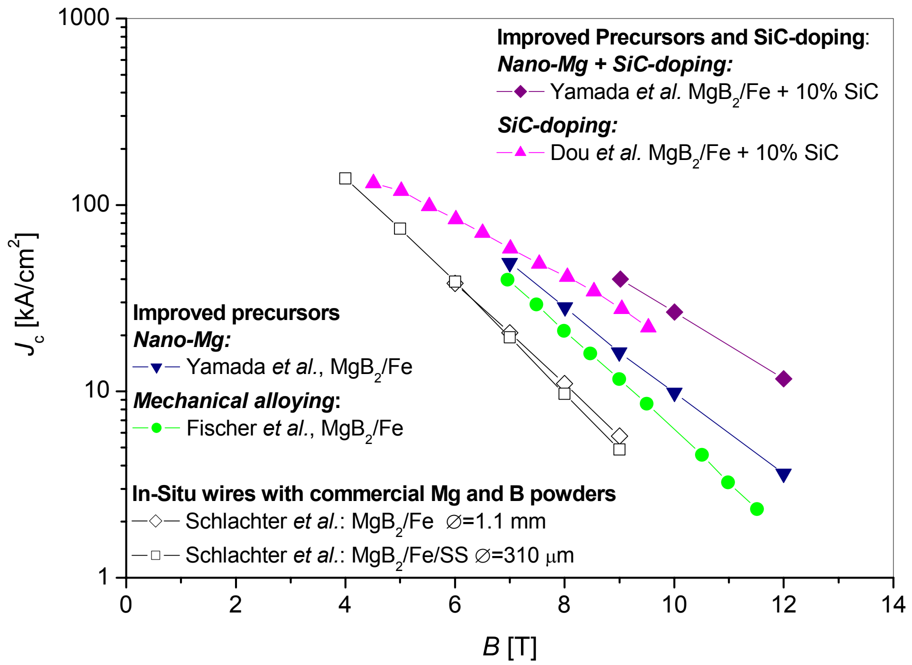

Fig. 12: $J_c(B)$ of a MgB_2/Fe wire (Ø = 1.1 mm) and a MgB_2/Fe/SS wire (Ø = 310 µm) made from commercial Mg and Boron powders without doping (this work) in comparison to $J_c(B)$ of MgB_2/Fe wires with improved precursors. The improvement of precursors and resulting increase of J_c especially in high magnetic fields was achieved by mechanical alloying (Fischer *et al.* [21]), use of Mg powder with nano grain size (Yamada *et al.* [22]), and SiC-doping (Dou *et al.* [11] and Yamada *et al.* [22]).

considered. Nevertheless if the bottlenecks of the PIT-route are overcome and reliable techniques for the industrial production of technical multifilamentary MgB_2 wires are achieved with a performance of the actual best results for the current densities and critical fields, MgB_2 conductors might come into economical competition with NbTi for use at 4.2 K or even for operation close to 20 K. The most successful preparation route actually, MgB_2 doped with 10% nanosize SiC, is already in work for the long length industrial process [27].

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