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2009 Supercond. Sci. Technol. 22 105017

(http://iopscience.iop.org/0953-2048/22/10/105017)

Supercond. Sci. Technol. **22** (2009) 105017 (6pp) doi:10.1088/0953-2048/22/10/105017

Fabrication and superconducting properties of multifilamentary MgB_2 conductors for AC purposes: twisted tapes and wires with very thin filaments

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Received 7 July 2009, in final form 31 August 2009
Published 23 September 2009
Online at stacks.iop.org/SUST/22/105017

Abstract
In order to use MgB_2 conductors for AC applications, research and development efforts have to be carried out on their architecture and sheath material to minimize the AC losses. This paper will present the fabrication and characterization of two types of *ex situ* powder-in-tube processed pure MgB_2 conductors with properties making them good candidates for AC industrial applications: a multifilamentary tape with 12 filaments with a twisting pitch down to 17 mm and a 361-filament wire with an average single-filament size of about 30 μm. Concerning the twisted tapes we will present values of critical current density of about 10^5 A cm^{-2} at 4.2 K and 2 T and we will show that it is possible to achieve a proper compromise between the tape size, the twisting pitch length and the critical current density to face a reduction of the critical current density as a consequence of the strain on the filaments. Concerning the 361-multifilamentary wire we will show appreciable values of critical current density of about 5×10^4 A cm^{-2} at 4.2 K and 2.5 T which, together with the advantages given by the high number of very thin filaments and the non-magnetic matrix, could justify their employment.

1. Introduction

The rapid development and improvement of MgB_2 wires and tapes have recently allowed the employment of this superconducting material in industrial DC applications such as low field MRI magnets [1]. Several papers have also been published on the applicability of the MgB_2 conductors in AC devices such as SFCL [2] and transformers [3]. Nevertheless, further efforts are still needed to develop and optimize these conductors in terms of performance in the AC regime, in particular in facing the losses issue. Therefore the research work should be focused on the development of multifilamentary wires or tapes with a large number of fine and possibly twisted filaments. Furthermore a non-magnetic and high resistivity metal sheath is preferable in order to minimize the contribution to the AC losses of the magnetic hysteresis and the eddy currents in the matrix. Some publications treating this kind of MgB_2 wire development have appeared [4–6], while other groups experimented with the fabrication of multi-strand MgB_2 cables [7–9]. The highest number of filaments reported is 61 [5], while in [4] a minimum filament size of about 75 μm was reached. In all these cases, the *in situ* powder-in-tube (PIT) technique was employed to realize the multifilamentary MgB_2 conductors.

The aim of this work is to show the realization of twisted multifilamentary MgB_2 wires with a non-magnetic sheath and with a larger number of finer filaments by the *ex situ* PIT technique and their characterization in terms of transport properties. In particular, we will show the behaviour of a stabilized 12-filament wire with a twisting pitch down to 17 mm and of a wire with 361 filaments whose average size is about 30 μm.

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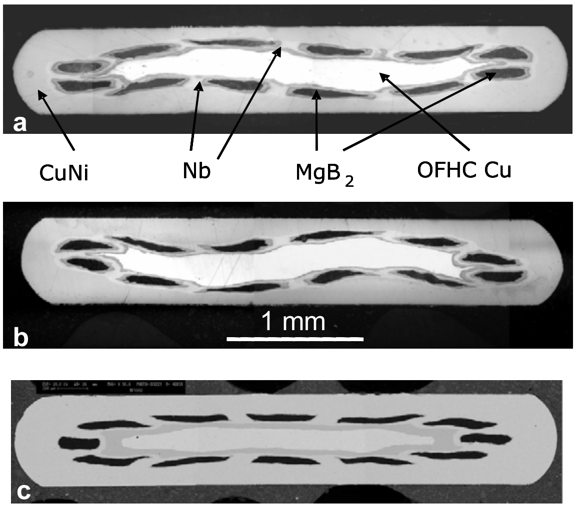

Figure 1. Cross section of the CuNi (a), CuNi_t70 (b) and Columbus tape (CT) (c) samples showing the same architecture.

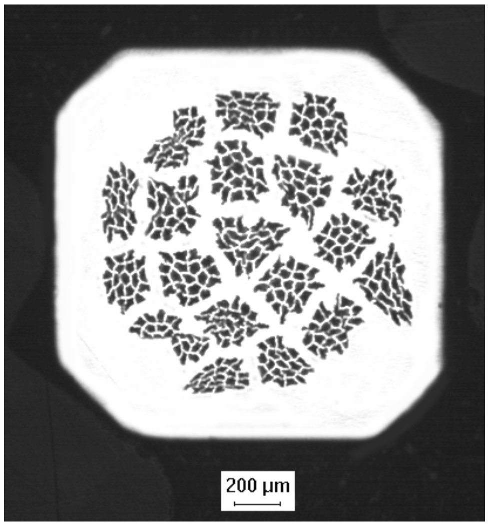

Figure 2. Micrography of the cross section of the unsintered CuNi361 sample.

2. Experimental details

MgB_2 multifilamentary wires were realized by the *ex situ* PIT method [10]. To prepare the pre-reacted pure MgB_2 powders a mixture of Mg (99.99%) and commercial amorphous B (99%) with a molar ratio of 1:2 was heat-treated at 760 °C for 1 h in an Ar/5%H_2 gas-mixture atmosphere to avoid possible oxidation of magnesium during the reaction and minimize the amorphous layer containing MgO around the MgB_2 grains [11]. A tube of Nb with an outer diameter of 13 mm and a wall thickness of 1 mm was inserted into a further tube of cupronickel (Cu70Ni30) alloy with an outer diameter (OD) of 16 mm and a wall thickness of 1.5 mm. This composite tube was filled by the pure MgB_2 powders. The function of the Nb barrier was to avoid the MgB_2 poisoning by the cupronickel sheath during the final heat treatment. The composite was drawn into a wire of about 3.5 mm OD. Twelve pieces of this wire were inserted in a monel (Cu30Ni70 alloy) tube in such a way to form a sort of crown around an inner part made of oxygen-free high conductivity copper (OFHC Cu) surrounded by an Nb chemical barrier. The role of OFHC Cu was to thermally and electrically stabilize the final conductor. The further Nb barrier prevents Ni from diffusing and contaminating the Cu stabilizer. The monel has been chosen as an external sheath because, being harder than cupronickel, it allows a better and more uniform cold deformation of the inner filaments. The so-prepared composite conductor was groove rolled and drawn to a round wire with a diameter of 2 mm. In this step a piece was flat rolled, obtaining a tape 3.6 mm wide and 0.65 mm thick, whereas the other two pieces were twisted with different pitches and subsequently flat rolled, obtaining two tapes with the same width and thickness defined above and a final twisting pitch of 130 and 70 mm, respectively. All samples were heat-treated at 965 °C for 4 min in Ar/5%H_2 flow by a continuous heat treatment apparatus.

In order to test the possible contribution to the AC losses of the metallic sheaths, a magnetization measurement versus magnetic field was performed by a commercial MPMS Quantum Design SQUID magnetometer on this sample at 40 K. Neither cupronickel nor monel alloys showed any magnetic hysteresis.

Table 1. Twisting properties of the multifilamentary wire.

Sample	Twist pitch (mm)
CuNi	Not-twisted
CuNi_t130	130
CuNi_t70	70

In table 1 the properties of the samples are summarized and in figure 1 the cross sections of the not-twisted and 70 mm twisted samples are shown. The cross section of the tape produced by Columbus Superconductors S.p.A. [10] is shown for comparison.

To realize the multifilamentary wire with 361 filaments, 19 pieces of the MgB_2/Nb/Cu70Ni30 monofilament were inserted in a further cupronickel tube. The composite was groove rolled and drawn to a wire with a diameter of 3.5 mm. It was cut again into 19 pieces which were restacked in a monel tube. After the usual cold working a 1.75×1.75 mm^2 wire with 361 thin filaments was obtained. The cross section of this sample (CuNi361) before any heat treatment is shown in figure 2. With the Nb barrier around the MgB_2 filaments being very thin at this stage of preparation, several final heat treatments at different temperatures were performed in order to check the possible chemical reaction between the Cu of the sheath and the superconducting phase. The results will be discussed in section 3.

Transport critical current measurements were performed by the four-probe voltage/current method using the 1 μV cm^{-1} criterion. The measurements were carried out at 4.2 K on 10 cm long samples in a bath cryostat and in an applied magnetic field up to 13 T at Grenoble High Magnetic Field Laboratory. The analysis on the cross section of the samples were performed by a scanning electron microscope (SEM) equipped with EDS for energy dispersive x-ray analysis.

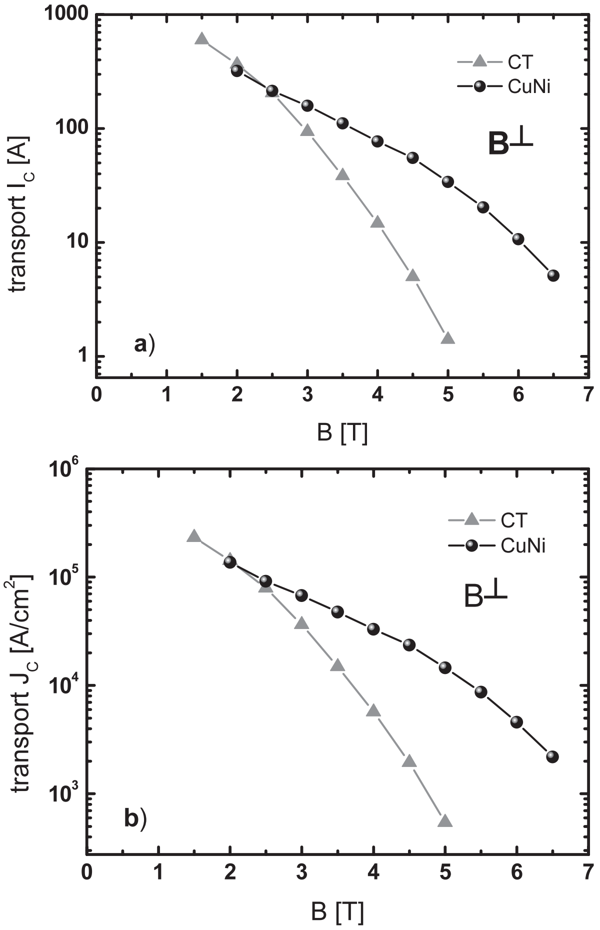

Figure 3. Transport $I_C(H)$ (a) and $J_C(H)$ (b) of the CuNi and CT samples at 4.2 K.

3. Results and discussion

3.1. Twelve-filament twisted tape

As is possible to see in figures 1(a) and (b), concerning the composite structure of the samples, there are no differences given by the twisting process. In particular, the size and disposition of the filaments are the same as well as the size of the copper and niobium barriers. The filling factor of both tapes is about 9% and the copper amount is 0.48 mm^2, about 20% of the whole tape cross section.

The transport critical current behaviour in a magnetic field, $I_C(H)$, of the CuNi tape in a perpendicular configuration with respect to the field direction was measured and compared with that of a commercial stabilized MgB_2 tape (CT) with the same configuration produced by Columbus Superconductors S.p.A. The width and the thickness of this tape are equal to those of the CuNi sample and the architecture is similar. The differences are that [10] in the CT sample the sheath is nickel and the barrier around the OFHC copper is iron; both are magnetic materials. The filling factor is about 11% whereas the copper amount useful for the stabilization is about 0.26 mm^2. Finally there is no niobium barrier. The undoped MgB_2 powders used for the CT tape were synthesized at 910 °C for 1 h in Ar/5%H_2. Figures 3(a) and (b) show the $I_C(H)$ and $J_C(H)$ respectively

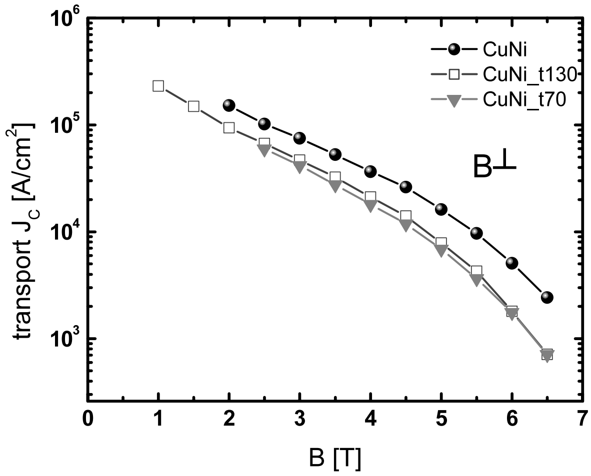

Figure 4. Comparison between the $J_C(H)$ of the CuNi, CuNi_t130 and CuNi_t70 samples.

at 4.2 K for these two tapes. The CuNi sample shows a better behaviour of the transport properties in a magnetic field, in particular at fields above 2 T. Taking into account that the powders used in CuNi were synthesized at 760 °C and those of the CT at 910 °C, this result agrees with that described in our previous paper [12]. It is fair to remark that the CT is a sample of a commercial 1.7 km long tape with a certified uniformity of the superconducting properties, while the CuNi is a short sample produced in the laboratory.

We would still like to point out that, with the cross section being the same, in the CuNi tape the copper amount was twice the CT one: from an application point of view, in particular regarding the stabilization issue, this is a very remarkable advantage.

In figure 4 the $J_C(H)$ for the CuNi_t130 and CuNi_t70 are shown. The CuNi $J_C(H)$ has been inserted for comparison. A J_C degradation of about 30% with respect to the CuNi has been observed for the CuNi_t130. This reduction of J_C could be ascribed to a decrease in grain connectivity. Indeed, during the twisting process the MgB_2 filaments, which are arranged around the Cu/Nb conductor core, undergo a certain degree of strain and thus lengthening, depending on the twist pitch length and on the diameter of the wire at the twisting stage: at larger diameters and shorter twist pitch corresponds a higher strain. In this case the wire diameter was 2 mm.

All this probably involves a loss of connectivity between the grains inside the filaments. Reducing the twist pitch down to 70 mm, J_C further decreased although only by 10%. Nevertheless a twist pitch as short as possible is preferable to minimize the AC losses.

To reduce the twist pitch avoiding a too large grain connectivity degradation, we tried to perform the twisting process of the wire at a smaller diameter stage. We produced new samples by twisting the wire with a diameter of 1.6 mm. After the usual flat rolling step we obtained two tapes with a final twist pitch of 30 mm (named CuNi_t30) and 17 mm (named CuNi_t17), respectively. In figure 5 the cross section of the CuNi_t30 is shown. The cross sections for these last two samples are equal. The configuration is very similar to those

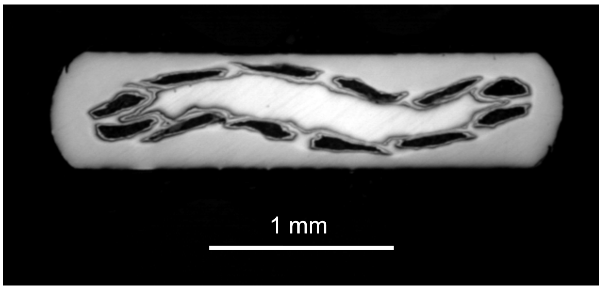

Figure 5. Cross section of the CuNi_t30 tape.

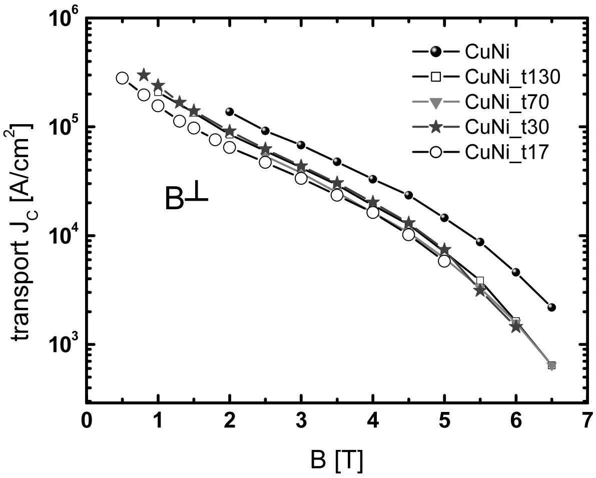

Figure 6. Comparison between the $J_C(H)$ of all twisted samples.

relative to CuNi and CuNi_t70 seen above. However, being that their wire diameter is smaller than that of the previous samples before the rolling step and being that the thicknesses are equal, the width is smaller and is about 2.8 mm. The filling factor is about 8%. In figure 6 the J_C behaviour in the field of the CuNi_t30 and CuNi_t17 is compared with that of the previous samples. We have obtained similar values on samples with a four times shorter twist pitch length. In particular, as is possible to see in the graph, the J_C curve of the CuNi_t30 nearly exactly retraces the CuNi_t130 one as well as the J_C curve of CuNi_t17 retraces the CuNi_t70 one. This should confirm that the observed degradation of the transport properties during the twisting process was due to the relative strain of the filaments. But against an useful shorter twist pitch, a reduction of the effective superconducting cross section of about 40% occurred: it will be necessary to take this into account during the design phase of a possible superconducting device.

However, these results suggest that, for each particular application, it is possible to achieve a proper compromise between wire size and twist pitch length in order to minimize the J_C degradation.

3.2. Multifilamentary wire with 361 very thin filaments

Figure 7 is an enlarged view of the cross section of the CuNi361 wire, showing MgB_2 filaments with an average size of about 30 μm. The Nb barrier around each filament is very thin and the filling factor is about 12%. We performed on it the usual heat treatment used for the tapes described above, i.e. at 965 °C for 4 min. In figures 8(a) and (b) the cross section after this heat treatment and the relative detail are shown.

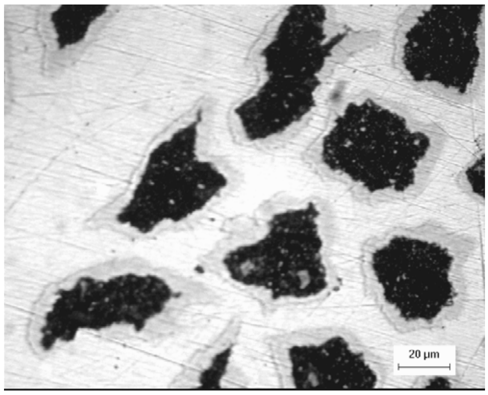

Figure 7. Detail of the unsintered CuNi361 cross section.

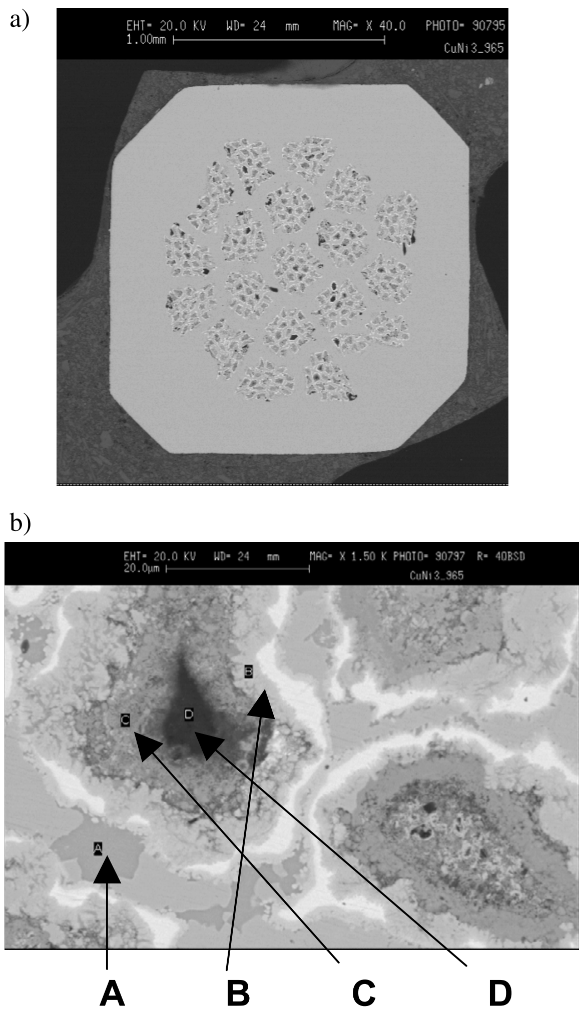

Figure 8. (a) SEM micrography of the CuNi361 cross section after the heat treatment at 965 °C, (b) detail of the sintered CuNi361: the different regions of the reaction between MgB_2 and the matrix are marked.

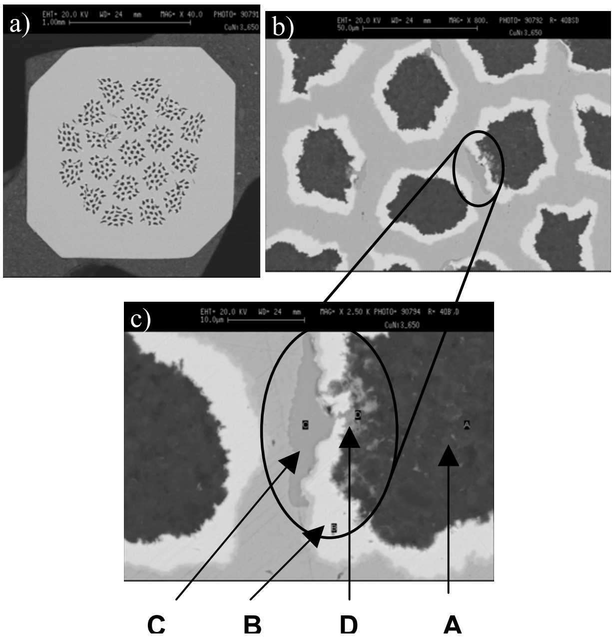

Figure 9. (a) SEM micrography of the CuNi361 cross section after the heat treatment at 650 °C; (b) and (c) details and analysis of the filaments.

It is evident that almost all of the MgB_2 filaments have reacted with the matrix and the superconducting phase was completely destroyed. From the energy dispersive x-ray analysis, four different regions of this reaction were identified. Referring to figure 8(b), in A the presence of Mg inside the matrix has been observed; in B and C, in the region previously occupied by MgB_2, there is the presence of Ni and Cu in different percentages: in B it is higher for Cu, whereas in C it is higher for Ni. In D there is still some MgB_2, while the light zones are residual parts of the Nb barriers.

Another heat treatment has been performed at a lower temperature with the aim to limit the damaging reaction and at the same time to sinter the MgB_2 phase enough to guarantee satisfying transport properties. The wire has therefore been heat-treated at 650 °C for one and half hours in Ar/5%H_2 flow. In figures 9(a)–(c) the cross section and its relative details are shown. The resulting filling factor was about 10%.

The filaments appear undamaged. Only where the thin Nb barrier was broken has a negligible undesired reaction been observed. Referring to figure 9(c), in A there is pure MgB_2 phase, in B the undamaged Nb barrier; the presence of Mg has been found in C as well as Cu and Ni detected in D.

In figure 10 the $I_C(H)$ curve and the relative J_C values are shown. As expected J_C is not as high as after a higher temperature sintering like, for example, for the CuNi sample. Even so, considering the advantages given by the high number of very thin filaments and the non-magnetic matrix in terms of stability and AC losses, it is reasonable to think that this kind of sample could be employed in low field applications, such as a superconducting fault current limiter (SFCL) or small superconductivity magnetic energy storage (SMES). Furthermore it is still needed and possible to optimize in terms of critical current the final heat treatment, investigating the temperature range between 650 and 965 °C.

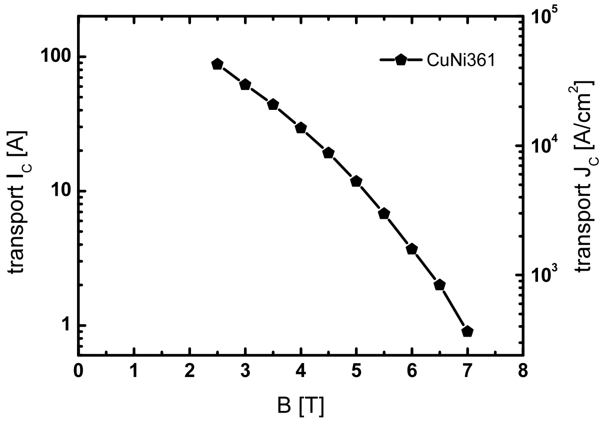

Figure 10. $I_C(H)$ and $J_C(H)$ curves for the CuNi361 wire heat-treated at 650 °C.

4. Conclusions

In summary we have successfully realized multifilamentary twisted stabilized MgB_2 tapes with a twisting pitch length down to 17 mm and a 361-filament wire with an average filament size of about 30 μm, the finest filaments obtained until now. Both conductors are endowed with a non-magnetic sheath. It has been therefore shown that it is possible to improve the MgB_2 AC performance in terms of loss reduction and thermal stability and thus enhance its application potential. Concerning the twisted tapes, it has been shown that, for each particular application, it is possible to achieve a proper compromise between the tape size, the twisting pitch length and the critical current density to face a reduction of the critical current density as a consequence of the strain on the filaments and their relative connectivity loss.

Concerning the 361-filament wire, although an optimization of its final heat treatment is still needed, we have shown that the obtained appreciable values of critical current density together with the advantages given by the high number of very thin filaments and the non-magnetic matrix could allow the employment of this kind of conductor in low field AC applications.

Acknowledgments

The authors wish to thank Dr Eric Mossang for assistance at GHMFL.

The support of the European Commission from the 7th Framework Programme Capacities ‘Transnational Access’, contract no. 228043-EuroMagNET II—Integrated Activities is acknowledged.

References

[1] Razeti M *et al* 2008 *IEEE Trans. Appl. Supercond.* **18** 882
[2] D’Alessandro R, Bocchi M, Rossi V and Martini L 2007 *IEEE Trans. Appl. Supercond.* **17** 1776

[3] Hascicek Y S *et al* 2009 *Supercond. Sci. Technol.* **22** 065002
[4] Schlachter S I *et al* 2007 *IEEE Trans. Appl. Supercond.* **17** 2842
[5] Sumption M D *et al* 2007 *IEEE Trans. Appl. Supercond.* **17** 2838
[6] Funaki K *et al* 2008 *J. Phys.: Conf. Ser.* **97** 012316
[7] Holúbek T, Schlachter S I and Goldacker W 2009 *Supercond. Sci. Technol.* **22** 055011
[8] Musenich R, Greco M, Razeti M and Tavilla G 2007 *Supercond. Sci. Technol.* **20** 235–8
[9] Kováč P, Hušek I and Melišek T 2008 *Supercond. Sci. Technol.* **21** 125003
[10] Braccini V, Nardelli D, Penco R and Grasso G 2007 *Physica* C **456** 209–17
[11] Vignolo M *et al* 2009 *IEEE Trans. Appl. Supercond.* **19** 2718
[12] Vignolo M *et al* 2008 *IEEE Trans. Appl. Supercond.* **18** 1175–8