The influence of the maximum heat treatment temperature on the performance of thin reinforced multifilament Bi-2212 wires for cables

R Nast, B Ringsdorf, B Runtsch, K-P Weiss, W Goldacker

Forschungszentrum Karlsruhe, Institut für Technische Physik, Postfach 3640, D-76021 Karlsruhe, Germany

E-mail: rainer.nast@itp.fzk.de

Abstract. For high field applications and applications operated at 20-30 K round Bi-2212 wires are still very interesting. For accelerator magnets or the future generation of fusion magnets operation currents up to 10 kA or even several 10 kA are required. For this purpose cabling of BSCCO wires in a low AC loss arrangement is necessary. With Bi-2212 wires optimized on \( J_c \) and \( T_c \) cabling to 3x3 and 7x7 strands was the goal, in order to demonstrate the feasibility of a cable-in-conduit concept. To reach the necessary mechanical stability for a cabling process a AgMg (2 at% Mg) outer sheath was used with a reinforcement twice of usual material. However, this sheath affects strongly the reaction kinetics, which leads to a decrease of the critical current density in comparison to a pure silver sheath. 7, 19 and 37 filament wires were fabricated by the powder-in-tube (PIT) and partial melting process, treated in pure oxygen. The current carrying capacity of the Bi-2212 wires essentially depends on the maximum heat treatment temperature \( T_{max} \) and the filament diameter. The best average \( J_c \) of 90 kA/cm² (4.2 K, self-field) was reached in 37 filament wires with filament sizes of 29 µm, treated at the maximum temperature between 879°C and 884°C. We realized stranded conductors and report first results of their superconducting properties.

1. Introduction

For practical applications like high field magnets at 20-30 K ceramic high temperature superconductors are the most promising materials due to the flat \( I_c(B) \) dependency. High-performance mechanically reinforced Ag-sheathed Bi\(_2\)Sr\(_2\)CaCu\(_2\)O\(_{8+d}\) (Bi-2212) tapes and wires are in development for this application.

In the past, several groups investigated Bi-2212 mono and multifilament tapes and wires [1-4] but all of them used pure Ag sheath without mechanical reinforcement. For large magnets as for instance in fusion facilities we need a high current conductor with extraordinary mechanical strength. To fulfill these requirements we fabricate mechanical reinforced round multifilament Bi-2212 wires with Ag tubes for the monocores and AgMg (2 at% Mg) as outer sheath. As an alternative to the Rutherford cable which was well developed at the LBNL [5] we followed the significant simpler concept of a cable-in-conduit conductor (CICC) which was already widely studied and fabricated for making toroidal field and poloidal field magnets in superconducting fusion facilities from LTS wires [6, 7]. This concept also provides a transposition of the strands for low AC losses. The difficulty in the
Rutherford cable approach is to deal with a huge $I_c$ degradation due to winding damage and chemical core poisoning. In our approach we use round Bi-2212 wires too because of their good cabling and winding behavior and the proven ability to carry high currents [8, 9].

The transport critical current density $J_c$ in wires depends strongly on the density of the superconducting core, and the presence of residual non-superconducting phases. Generally, melt processing was applied to get homogeneous dense filaments. In melt processing, the Bi-2212 is heated until it congruently melts, forming liquid and non-superconducting crystalline phases [10, 11]. During cooling, the superconducting 2212 phase forms from the liquid and crystalline phases in the melt. A significant difficulty with melt processing is that during cooling the Bi-2212 phase formation is often not complete, so remnant secondary phases remains in the fully processed conductor. These residual phases need to be eliminated to achieve a high $J_c$. Therefore the melt-solidification conditions have to be optimized. The commonly observed intergrowths between the filaments favor the overall connectivity in the superconducting part.

2. Experimental

For the preparation of Bi-2212 tapes we used commercial precursor powder with the stoichiometry Bi$_{2.17}$Sr$_{1.95}$Ca$_{0.89}$Cu$_{1.99}$O$_{8+d}$ supplied by Nexans SuperConductors GmbH. The tapes were prepared by the PIT technique using pure Ag tubes (8 mm outer diameter, 6 mm inner diameter). The powder was packed into these tubes and then deformed by swaging and drawing to monocore wires with a hexagonal cross-section for the bundling into an AgMg (2 at% Mg) tube with 12 mm outer diameter and 10 mm inner diameter as outer sheath. The multifilament bundle was deformed again by swaging and drawing to different final diameters of < 0.5 mm.

For the heat treatment, the wires were cut into pieces of 7 cm or 20 cm which were melt processed in flowing pure oxygen in a tubular furnace. The melt-processing schedule consisted of heating the samples to the maximum heat treatment temperature ($T_{max}$) with a heating rate of 300°C/h, holding for 12 min, cooling with 10°C/h to 840°C, holding for 48 h, then cooling to room temperature (furnace cooling). To investigate the relation between $J_c$ and $T_{max}$, all the other parameters in the schedule were kept constant. Figure 1 shows the heat treatment profile of the partial-melting process. We varied $T_{max}$ over a wide range between 868°C and 894°C.

![Figure 1. Heat treatment profile of the partial-melting process.](image-url)
The reacted single wires were assembled to 1x3, 3x3 and 7x7 strand conductors. The transposition lengths relate to Nb₃Sn conductors and are 35 / 55 mm for the 3x3 strand conductor and 55 / ∞ mm for the 7x7 strand conductor.

\( J_c \) was measured using a conventional four-probe method at 4.2 K in self-field. The field dependence of \( I_c \) was measured in the high-field conductor test facility FBI [12]. To investigate the microstructures of the wires, scanning electron microscopy (SEM) and optical microscopy were used.

3. Results and Discussion

3.1. Single wires

All investigated samples, independently of the wire cross section and filament number, show an almost constant critical current above 876-877°C over a wide temperature range of more than 10°C. This is extremely important for a reproducible, homogeneous production of wires in large lengths. A dramatically increased critical current (peak effect) within a few tenth of a degree at the beginning of melting the 2212 phase as was found by Matsumoto et al. [3] is only weakly pronounced. Rikel et al. [13] confirmed this behavior but, however, it is in contrast to earlier papers in which a pronounced peak effect was shown [2, 3]. For a better comparison with literature data and to see the influence of different wire diameters, the critical current density is shown via \( T_{max} \) in figure 2.

![Critical current density as a function of the maximum heat treatment temperature](image)

**Figure 2.** Critical current density as a function of the maximum heat treatment temperature \( T_{max} \).

The average current density of all conductors with 0.48 and/or 0.195 mm in diameter is 60 kA/cm² ± 10 kA/cm², excluding the higher \( J_c \) at the begin of melting the 2212 phase (877-878°C), which leads to current densities of 80 ± 5 kA/cm². An exception are the conductors with 0.293 mm in diameter.
Here a trend to higher current density values is shown. Within 879 and 886°C an average critical current density of 90 kA/cm² is reached, however a large variation of ± 10 kA/cm² exists. A reason may be due to slight mechanical defects occur in the wires during processing or to the varying amount of filament intergrowths. Taking the entire temperature range into account an average $J_c$ of 80 kA/cm² with a standard deviation of 14 kA/cm² is obtained. Apart from the fact, the increase of the critical current density within the plateau region of the 37 filament conductors ("cyan") to 90 kA/cm² is clearly seen. In order to represent the influence of the filament diameter on the critical current density more transparently, this dependence in the plateau region between 879°C and 886°C is presented in figure 3.

![Figure 3](image.png)

**Figure 3.** Critical current density in dependence on the filament diameter.

With decreasing filament diameter the critical current density increases slowly. Reaching a filament diameter of 29 µm a clear current density increase to 90 kA/cm² is seen. Decreasing the filament size further, $J_c$ decreases again rapidly (62 kA/cm² at 26 µm). The conductor seems to come to its deformation limit and is partly irreversibly damaged, decreasing $J_c$ rapidly as a consequence. The highest average critical current density of 90 kA/cm² is reached with a filament diameter of 29 µm. In the literature $J_c$ values of about 100 to 165 kA/cm² (4.2 K, self-field) in single wires are reported [1, 4, 14] and the optimum currents correspond with filament diameters of 11 µm [14].

The most probable reason for the reduced critical current density in our conductors is the use of an Ag-Mg outer sheath with high Mg content, which affects the phase formation. This was shown for Bi-2223 tapes [15, 16] and leads to a reduction of the critical current density of about 1/3. Such degradation is caused by the reaction between the Mg and the filaments, which induces the contamination and/or the formation of impurity phases in the filaments. An additional reason for the $J_c$ reduction may be the reduced amount of intergrowths (figure 4) compared to [3, 14].
3.2. Stranded conductors

In order to show the possibility of using Bi-2212 wires for cable-in-conduit composites, we realized a 3x3 (twist pitch 35 / 55 mm) and 7x7 (twist pitch 55 / ∞ mm) strand cabling (length 20 cm). Figure 5 shows the cabled conductors.

![Figure 5. 3x3 (d_single wire = 0.48 mm, 19 fil, T_{max} = 883°C) and 7x7 (T_{max} = 881°C).](image)

In figure 6 the critical current $I_c$ of a single wire, a 3 stranded wire, the 3x3 strand and 7x7 strand cable as a function of an external magnetic field $B$ is shown. $B$ is perpendicular to the wire length and causes a degradation of the critical current at 10 to 12 T of about 30 % of the initial value. This corresponds to the behavior of pure silver wires. The n-value decreases in self field from 18 in single wires to 8 in the 3x3 strand cable. That’s a typical feature of such wires, corresponds to the behavior of Nb₃Sn wires and can be explained by the following fact. The current rearrangement caused by the inhomogeneities of $I_c$ in the single wires widens the superconducting transition and thus leads to a reduced n-value.
Figure 6. $I_c$ normalized on a single wire in dependence on the external magnetic field $B$ for a single wire, a 3 stranded wire, the 3x3 and 7x7 strand cable (insert: measured $I_c$ values).

Figure 6 indicates further that the $I_c$-$B$ relations of all the investigated samples normalized to a single wire are similar. So the behavior of a stranded cable is comparable with the performance of the single wires. This is an important prerequisite for the production of stranded cables with high strand numbers and a full transposition design. The self field effect on $I_c$ is also illustrated and is suppressed at an external applied magnetic field. The $I_c$ of the 7x7 strand cable could not be measured below 2 T because of the limitation of the current source to 350 A.

Figure 7 shows the $I_c$ dependence on the mechanical strain. Only above a strain of 0.5% a clear degradation of $I_c$ is shown. This is an improved very high value caused by the Ag-Mg outer sheath with increased Mg-content. Compared to a pure silver sheath the strain increases by a factor of 2.5.

Figure 7. $I_c$ as a function of the strain $\varepsilon$ for the 3x3 strand cable.
4. Summary
Thin Bi-2212-Ag/AgMg multifilament wires with 0.48, 0.293 and 0.195 mm in diameter were made and their \( I_c/J_c \) in dependence on the maximum heat treatment temperature was optimized. The highest average critical current density of 90 kA/cm\(^2\) was reached in 37 filament wires with 29 µm filament diameter. The reduced critical current density compared to literature data and the changed phase dimension is caused by the mechanical reinforced outer sheath of Ag-2 at% Mg. Therefore a substantial further increase of the critical current density is not to be expected upon further optimization. The large \( L_c/J_c \) plateau of 11°C (879-890°C) within the critical current remains constant favors an industrial production of the conductors, without a degradation of the critical parameters. The stranded conductors show a promising behavior for this kind of cable design. In a field of 10 or 12 T the conductors carry still 30 % of the current in self-field. The n-value decreases from 18 in the single wire to approx. 8 in the stranded 3x3 cable in self-field. This is caused by \( I_c \) inhomogeneities of the individual wires. As expected the \( I_c \) of all the stranded conductors is the sum of the \( I_c \) of the single wires. The Ag-Mg outer sheath causes the high compatibility against mechanical strains. Only above 0.5 % a clear degradation of \( I_c \) is visible.

The reinforced sheath, the homogeneous microstructure and the large plateau region of \( I_c/J_c \) are promising aspects for an industrial production and the use of stranded conductors in much larger scaled-up cable arrangements for magnet coils.

Acknowledgments
This work was supported in parts by the fusion program of the Helmholtzgemeinschaft HGF.

References
[1] Zhang W, Goodilin E A and Hellstrom E E 1996 Supercond. Sci. Technol. 9 211
[2] Polak M, Zhang W, Polynskii A, Pashitski A, Hellstrom E E and Larbalestier D C 1997 IEEE Trans. Appl. Supercond. 7 1537
[3] Matsumoto A, Kitaguchi H, Kumakura H, Nishioka J and Hasegawa T 2004 Supercond. Sci. Technol. 17 989
[4] Bigansolli A R, Cursino E, Santos F A and Rodrigues Jr D 2006 Proc. EUCAS’05 (Vienna, Austria, 11-15 Sept. 2005) J. Phys. Conf. Ser. 43 p 67, online at jpcs.iop.org
[5] Collings E W, Sumption M D, Scanlan R M, Dieterich D R, Motowidlo L R, Sokolowski R S, Aoki Y and Hasegawa T 1999 Supercond. Sci. Technol. 12 87
[6] Wang Q, Weng P and He M 2004 Cryogenics 44 81
[7] Lee S, Jeong S and Choi S-M 2008 Physica C 468 417
[8] Motowidlo L R, Galinski G, Ozeryansky G, Zhang W, Hellstrom E E, Sumption M and Collings T 1995 IEEE Trans. Appl. Supercond. 5 1162
[9] Hasegawa T, Koizumi T, Hikichi Y, Nakatsu T, Scanlan R M, Hirano N and Nagaya S 2002 IEEE Trans. Appl. Supercond. 12 1136
[10] Oka Y, Yamamoto N, Tomii Y, Kitaguchi H, Oda K and Takada J 1989 Japan. J. Appl. Phys. 28 L801
[11] Heine K, Tenbrink J and Thöner M 1989 Appl. Phys. Lett. 55 2441
[12] Specking W, Nyilas A, Klemm M, Kling A and Flükiger R 1990 Proc. MT-11 (Tsukuba, Japan, 28 Aug.-1 Sept. 1989) p 1009
[13] Rikel M O, Arsac S, Soileux E, Ehrenberg J, Bock J, Marken K, Miao H, Bruzek C-E, Pavard S, Matsumoto A, Hellstrom E E and Motowidlo L 2006 Proc. EUCAS’05 (Vienna, Austria, 11-15 Sept. 2005) J. Phys. Conf. Ser. 43 p 51, online at jpcs.iop.org
[14] Motowidlo L R, Galinski G, Ozeryansky G, Zhang W and Hellstrom E E 1994 Appl. Phys. Lett. 65 2731
[15] Goldacker W, Mossang E, Quilitz M and Rikel M 1997 IEEE Trans. Appl. Supercond. 7 1407
[16] Quilitz M and Goldacker W 1998 Supercond. Sci. Technol. 11 577