INVESTIGATION ON TECHNICAL SUPERCONDUCTORS FOR LARGE MAGNET SYSTEMS IN BOCHVAR INSTITUTE

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Abstract

The materials science of technical superconductors in USSR and later in Russia has been concentrated in Bochvar Institute of Inorganic Materials. This paper reviews the main activities of Bochvar Institute in design and development of the NbTi, NbTiTa and Nb_3Sn superconductors. The critical features of design and factors of manufacturing processes determining the superconducting properties of the technical NbTi and Nb_3Sn superconductors are analyzed. The data on tantalum influence on the NbTi wires superconducting properties is given. The examples of NbTi wires designed for large projects such as accelerator (UNK), tokamaks (T-7 and ITER) are presented. The comparison of bronze processed and internal tin Nb_3Sn wires has been done. The properties of the Nb_3Sn wires designed and fabricated for application in toroidal field coil insert and toroidal field coils of ITER magnet system are discussed. The examples of Nb_3Sn wires strengthened by the microcomposite Cu-Nb alloy are presented. The perspectives of the further increase of superconducting properties for both types of Nb_3Sn wires have been estimated.

1. INTRODUCTION

Bochvar Institute of Inorganic Materials (BI) has begun the investigations on the technical superconducting wires in late 1960-s. For shortening the transfer of the designed manufacturing processes from laboratory to the industry, industrial scale equipment was installed in the experimental shop of BI. In this way, full-scale pilot batches of superconducting wires could be produced just after the initial development and testing of designed laboratory samples. The primary objectives were the development of winding wires for the first tokamak in the world with superconducting magnet system (T-7) [1] and for the magnet system of the Serpukhov's accelerator (UNK). Both tasks have been completed successfully [2]. An industrial production of superconducting wires in USSR was then established in 1970-s.

The superconducting wires for many other applications were also designed, including NbTi wires for MRI tomographs, generators, for persistent current switches, for different types of scientific devices – wigglers, gyrotrons etc. [3-6].

Work on Nb_3Sn superconductors was initiated practically at the same time, beginning with the tape conductors produced by liquid phase reaction of the Zr doped Nb with Sn-Cu melt [7]. In the beginning of 1970-s the development of multifilamentary Nb_3Sn superconductors by bronze process and internal tin process was started.

For Tokamak-15 [8], a force-flow cooled conductor (340 m long, 17.4mm by 6.5mm in cross section) consisting of Nb_3Sn-based superconducting transposed wire with 11 individual bronze processed nonstabilized strands (1.5 mm in diameter), two copper tubes (4.0 mm in external diameter, 0.5 mm in wall thickness), and a 1.2 mm thick electrolytic copper layer incorporating them (Fig.1) was developed [9]. The critical current of such a current-carrying element was larger than 8.5 kA in a field of 8 T. Approximately 90 t of conductor were produced in an industrial way, requesting the production of more than 25 tons of wires. The TF coils were produced by the react and wind method. The positive experience of the use of Nb_3Sn wires in large magnets opened way for further development of even larger Nb_3Sn based magnet systems.

At present, the progress in High Energy Physics and Fusion relies on the successful realization of currently running Projects such as the LHC and ITER. Magnet systems of both facilities consume around 1000 tons of NbTi and Nb_3Sn superconductors each. Therefore the question of cost is one of the important factors altogether with performance parameters. Next generation of accelerators and fusion reactors will require higher magnetic fields (in the range of 12-16 T), which is a challenge for currently existing commercially available superconductors. The possible superconductor candidates for high field (14-16 T) magnetic systems are YBCO, Bi2212, Nb_3Al, Nb_3Sn (Fig.2).

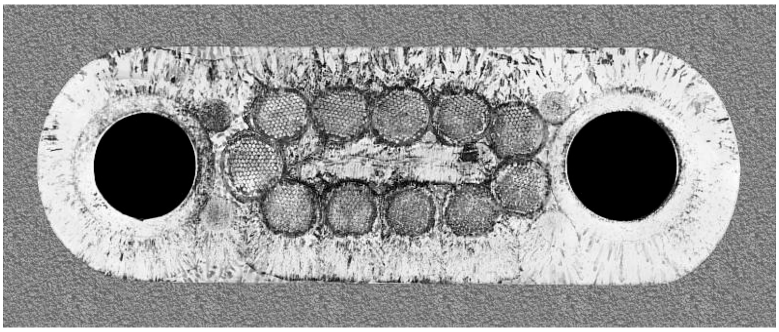

Figure 1. Cross section of force-flow cooled Nb_3Sn conductor for toroidal field coils of T-15

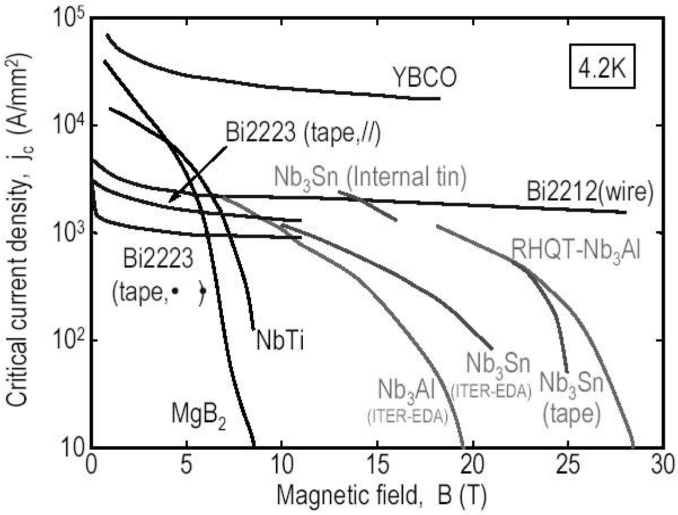

Figure 2. The schematic comparison of current-magnetic field properties for different contemporary superconductors [10,11].

Taking into account the abovementioned cost factor, which practically eliminates the high temperature superconductors, it could be stated that for the nearest future the choice of wires is limited to Nb_3Al and Nb_3Sn, both low temperature superconductors.

2. INVESTIGATION ON NBTI WIRES

The NbTi wires due to excellent mechanical properties and low cost were and still remain the workhorse for the magnetic fields up to 8 T. The main objectives of the initial R&D program were to develop NbTi multifilamentary wires with higher critical current density (Jc), lower losses or finer filaments, longer piece lengths, higher wire uniformity and greater yields. One of the crucial problems was to avoid significant filament sausaging (the variation in filament diameter along the length of the wire). The most important factor influencing on sausaging was the formation of brittle Cu-Nb-Ti intermetallic inclusions at the superconductor-Cu interface. These inclusions do not deform uniformly with the filament during wire drawing, leading to filament sausaging. Introduction of Nb diffusion barrier is now widely used to prevent intermetallic inclusions formation [6].

The nature of deformation of BCC NbTi filaments in FCC Cu matrix by cold drawing also could lead to sausaging through texturing. During the deformation the texture of drawing with direction of z = [011] is developing in NbTi filaments. In BCC NbTi the dislocations slipping system [111](011) is prevailing, therefore (due to the texturing) less than 5 active slipping systems remain. In this case a uniform deformation is substituted by a plane deformation and instead of obtaining cylindrical filaments, the finale shape is ribbon like. To mitigate this effect the local volume fraction of NbTi should be properly chosen through nonuniform arrangement of filaments in the strands cross section [12].

The abovementioned major problems were resolved in the late 70-s and reliable technical NbTi wires with high critical current became commercially available. In 1978 the first tokamak in the world with superconducting magnetic system T-7 has been successfully built and tested. For the Russian accelerator program UNK, 120 tons of NbTi wires with 8910 filaments (6μm in diameter) and critical current density up to 2600 A/mm^2 have been produced. Later on, critical current density up to 3000 A/mm^2 (5T, 4.2K) in commercially produced wires has been attained.

Today the limit of accelerator magnets wound from binary NbTi conductors and operated in superfluid helium at 1.9 K lies in the 10-to-10.5T range. It is possible to raise Bc2 of NbTi by addition of Ta. In Bochvar Institute two 4620 filamentary strands were designed and fabricated on the base of ternary NbTiTa alloys with 12 and 20 wt.% Ta (whose layout meets the LHC specifications) [13-17]. The main efforts were focused on the preparation of highly homogeneous alloys and the optimization of heat treatments to achieve high Jc at high fields. At 2K maximum Jc for NbTi-20wt%Ta strand reached 1550 A/mm^2 at 10 T and 750 A/mm^2 at 12 T. At 1.85K Jc is increased by 20% attaining 450 A/mm^2 at 13 T which was about twice higher than that obtained in NbTi strands.

In the framework of the ITER Project the RF Participant Team has manufactured the NbTi Cable, and shipped it to EFDA for further fabrication of poloidal field insert coil (PFCI). The testing of PFCI is planned to be carried out in Japan during 2004. The wire was designed and produced in Bochvar Institute in an amount of 400 kg and cabling was performed at the Cable Institute. The cross section of this wire is presented in Fig.3. The parameters of this wire were as follow: Wire diameter of 0.73 mm; Number of filaments 2346; Filament diameter 9.8 μm; Cu/non Cu Ratio 1.4; Critical current density (by specification) > 2700 A/mm^2 (5T, 4.2K); (2800-2900 A/mm^2 – measured values). The fabrication of the wire was carried out from composite billets of intermediate size because of the relatively small amount of

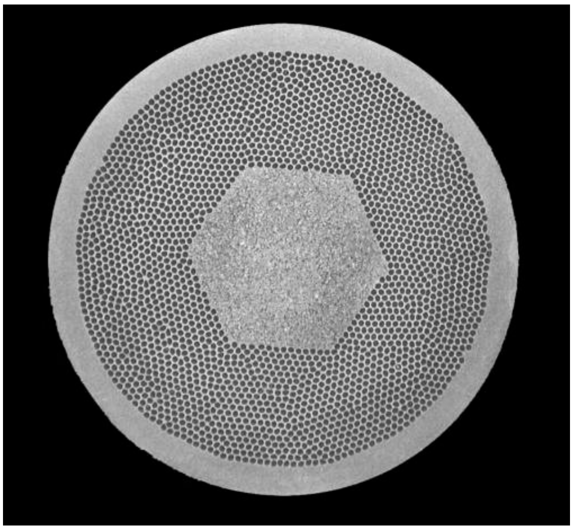

Figure 3. NbTi wire designed and produced for fabrication of ITER experimental poloidal field coil-insert (PFCI);

wire was needed [18]. That is why the critical current density of these wires was specified to be slightly less than it should be in the final wires for the PF coils.

In the course of preparation for the PFCI testing some benchmarking measurements were undertaken. The Bochvar Institute characterized the strands for PFCI in a wide range of temperature and magnetic fields. The same work has been carried out in CEA (France). Comparison of CEA results with those of BI (defined as VNIINM in Fig.4) was done for the five values of B explored.

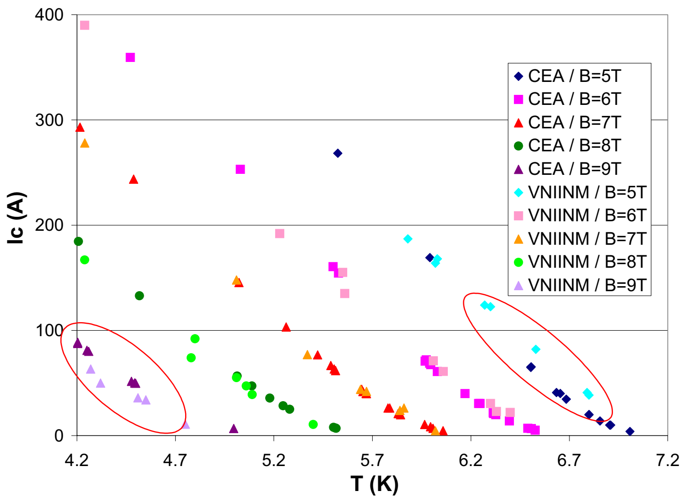

Figure 4. Temperature dependencies of PFCI NbTi strands critical currents for different magnetic fields, measured in BI (VNIINM) and CEA.

For the major part of the data compared a satisfactory consistence between CEA and BI measurements was obtained except for “extreme” magnetic field values (5T and 9T). In those cases, CEA results are found lower than VNIINM ones at low fields and higher at high field. This is probably hinting at differences in the structure of the tested strands (filaments structure, impurities ratio…).

NbTi wires designed for the ITER PF coils production are presented in Fig.5. In the process of preparation for large scale production of these strands the composite billets were extruded from 250 mm in dia. In accordance with specification Jc should be higher than 2900A/mm^2 with a filament diameter of 6 μm; Cu/SC ratio 1.4 (for the coils PF1 and PF6) [19].

Model fine filament NbTi 0.65 mm wire, intended for operating in fields having sweep rate from 1 up to 4 T/s, has been developed and manufactured in Bochvar Institute (Fig.6) [20]. The wire was fabricated by a single stacking method. Each filament (3.46 μm in dia) was surrounded by a matrix of

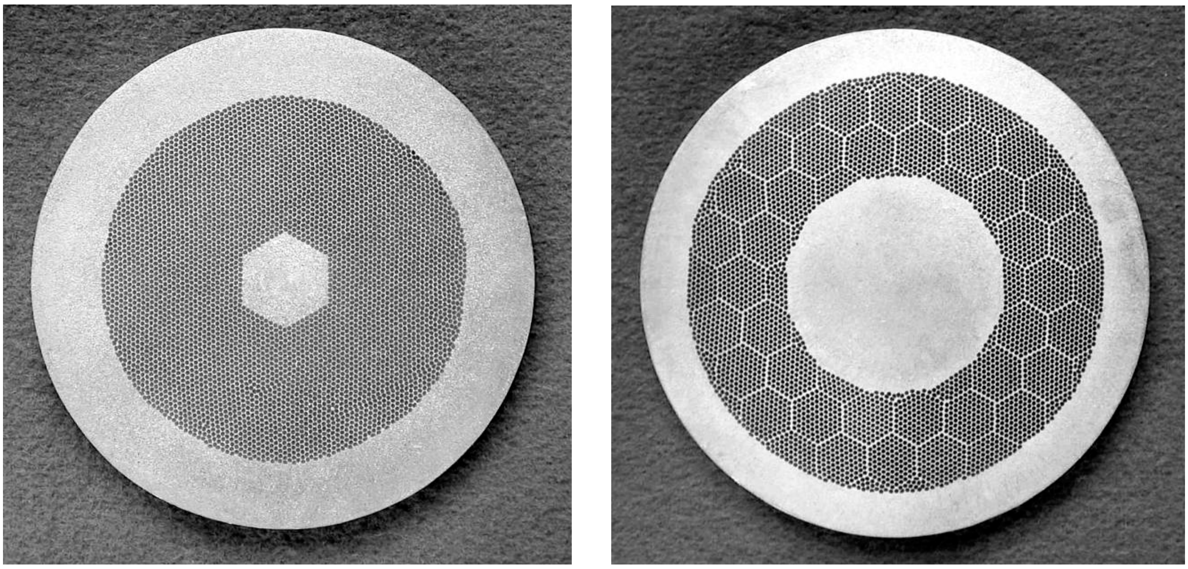

Figure 5. Cross sections of NbTi wires designed for ITER PF1 and PF6 coils.

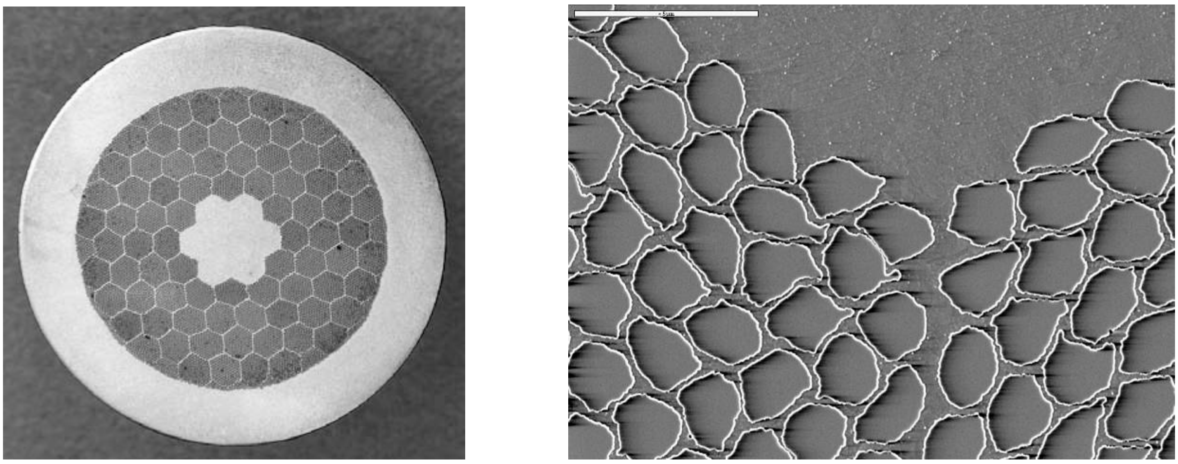

Figure 6. Cross section (a) and fragment of structure (b) of the NbTi wire designed for the application in the magnet systems with fast sweep rate up to 4 T/s.

commercial MN-5 alloy (Cu-5wt.%Ni). The spacing is 0.5 μm. The Cu/non Cu ratio is 1.8. Every 37 NbTi/Nb/CuNi rods were inserted into hexagonal copper tube made from high purity Cu.

The filament zone was arranged in cross section in such a way that the central copper core occupies about 7 % of area. The central copper core, tubes and the external sheath were fabricated from high conductivity copper with (R^{273}/R^{10}) > 250. The critical current density of this strand was higher than 2900 A/mm^2 (5T, 4.2K). A low hysteresis loss values of 51 kJ/m^3 per wire and 144 kJ/m^3 per superconductor volume have been achieved.

3. INVESTIGATION ON NB_3SN WIRES – DESIGN AND PROPERTIES

Two well established viable technologies are generally used for commercial production of Nb_3Sn strands – the so called bronze process and internal tin process. Due to some peculiar differences in these methods the process of optimization could be different, but principal approaches have a lot in common. Critical currents in Nb_3Sn strand depend on the volume fraction of the superconducting phase and the pinning of the magnetic flux vortices on lattice defects, the critical temperature (T_c), and the upper critical field (B_{c2}).

3.1 Bronze processed Nb_3Sn wires

Concerning bronze process two main features should be taken into account:

Disadavantages: Coprocessing of bronze matrix and niobium filaments during all the steps of the manufacture requires numerous intermediate annealing steps due to high rate of deformation hardening and as a consequence limited ductility of the Sn rich bronze.

Advantages: Extrusion of large composite billet is possible at the initial stage of deformation. Good metallurgical bonding of all elements of composite billet is guaranteed.

In bronze processed wires the bronze matrix is prepared by melting Cu and Sn (up to ~ 14-16 %wt of Sn) obtaining an alloy, which is a solid solution on the base of copper. The bronze matrix Cu-16%Sn due to high tin content is a two-phase material with brittle inclusions of intermetallic Cu-Sn phase. The part of Cu-Sn phase diagram is presented in Fig.7a. In the Fig 7b and 7c the microstructures of Cu-16%Sn alloy in cast condition and after homogenization are shown. It is seen that the brittle Cu-Sn eutectoid inclusions are present in the bronze matrix even after the homogenizing heat treatment.

The residual content of tin in the bronze matrix after reaction heat treatment could be less than 0.5 %. Assuming stoichiometric composition of Nb_3Sn phase, the 25-26 % of the area inside the diffusion barrier should be occupied by Nb filaments. Therefore the maximum attainable volume fraction of Nb_3Sn phase is approximately 35% in the area inside the diffusion barrier (Taking into account 37 vol. % increase resulting from complete transformation of Nb into Nb_3Sn).

Taking this into account the requirement of J_{nc} > 800 A/mm^2 (standard 12 T, 4.2 K) assumes the attaining of critical current density in Nb_3Sn phase higher than approximately 2700 A/mm^2 for bronze processed wires, which is not a trivial task.

In the frame of ITER model coils program Bochvar Institute has designed and produced approximately 1 ton of strands for the TF Model Coil Insert conductor, which met the HP-2 specifications [21,22]. The parameters of the designed strand were as follow: Volume fraction of Cu of 60 %; Diameter of the strand 0.81 mm; Number of filaments 7225; Diameter of Nb_3Sn filaments of 2.5 μm, Critical current density Jc (12T, 4.2 K, non-Cu) > 550 A/mm^2; Hysteresis losses Qh (non-Cu; ±3 T) < 200 mJ/cm^3. The cross section of the strand is presented in the Fig.8.

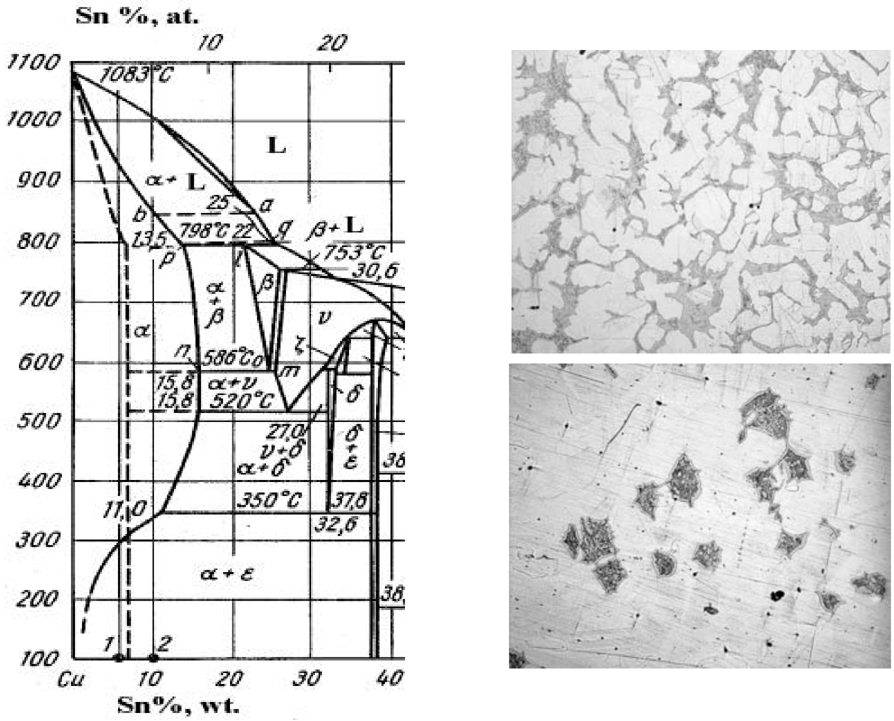

Figure 7. The Cu-rich part of Cu-Sn phase diagram (a) and microstructure of Cu-16%Sn alloy in cast state (b) and after the homogenizing heat treatment (c).

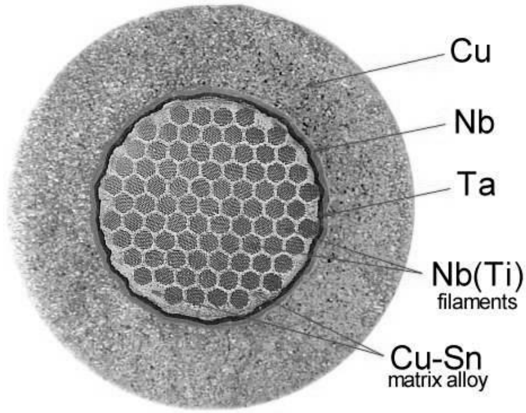

Figure 8. Cross section of the Nb_3Sn strand developed for PFCI in the frame of ITER model coil program.

The cabling and jacketing of the full size conductor were performed by Cable Institute, Coil has been wounded in Efremov Institute in St.Petersburg and successfully tested in JAERI (Japan) [23]. It is important to note that the spread of Jc values on samples heat treated with the TFCI and spreaded along the spiral height was less than 1% (average Jc = 579 A/mm^2) and there was almost no spread in the hysteresis loss values, which were lower than 200 mJ/cm^3[24].

Bronze processed Nb_3Sn wires with enhanced critical current properties have been developed in Bochvar Institute after the completion of the ITER model coil program. The cross section of one of the options is presented in the Fig.9 [25]. Critical current density of newly designed Nb_3Sn wires with 7851 filaments was equal to 774 A/mm^2 (12 T, 4.2 K); Hysteresis losses (+/-3T) of 337 mJ/cm^3; Cu/(non Cu) ratio of 1.2.

Another option of bronze processed Nb_3Sn wires with enhanced critical current properties is presented in Fig.10 [26]. The enhancement of critical current density has been achieved by the optimization of artificial titanium doping [27]. Each of the 12684 Nb composite filaments contained 4 Nb-Ti alloy rods (Fig.10b). The relationship between the amounts of Nb and Nb-Ti alloy has been chosen to be equal to 2 wt% of Ti in the filament after heat treatment.

The comparison of critical current carrying capabilities of the bronze processed wires designed in the framework of ITER project is given in the Fig.11. It was shown that bronze processed Nb_3Sn wires with low hysteresis losses could be produced with critical current density attaining 800 – 900 A/mm^2.

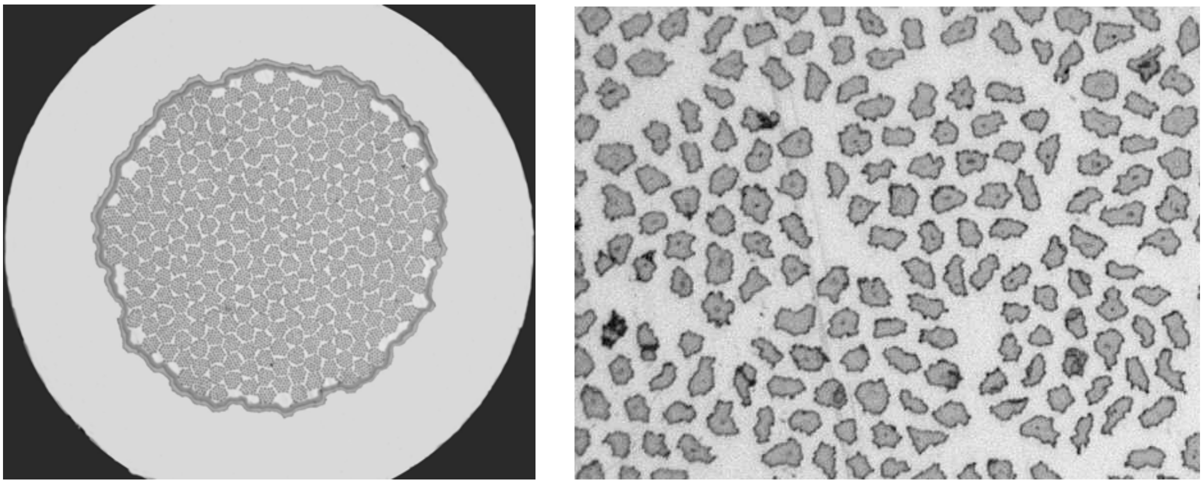

Figure 9. Cross section of the bronze processed Nb_3Sn wire with enhanced critical current density -(a) and the fragment of structure illustrating increased uniformity in the filaments arrangement -(b). ; Number of filaments 7851.

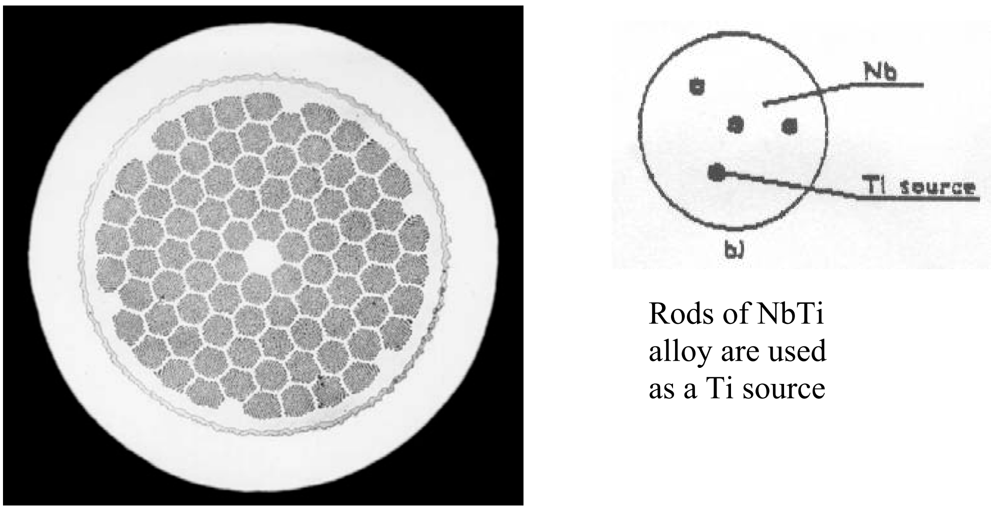

Figure 10. Cross section of the bronze processed Nb_3Sn wire with enhanced critical current density (a) and the fragment of structure illustrating the arrangement of Ti inserts inside the Nb filament (b).

Because the critical current carrying capability of superconducting wires is a structure dependent parameter, the investigations of microstructure including X-ray and electron microscopy methods are constantly under the way in Bochvar Institute as a basis for their development and optimization. Some examples of the Nb_3Sn grains microstructure in bronze processed wires, designed in the frame of ITER project are given in the Fig.12 [28].

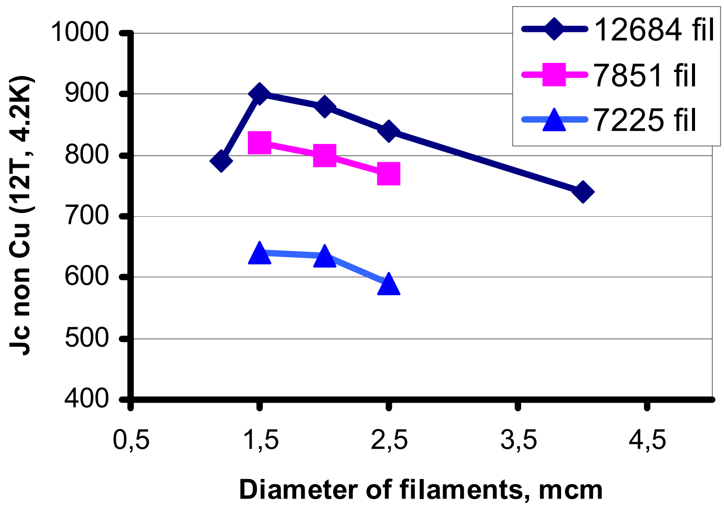

Figure 11. Critical current (non Cu, 12 T, 4.2 K) of bronze process Nb_3Sn wires.

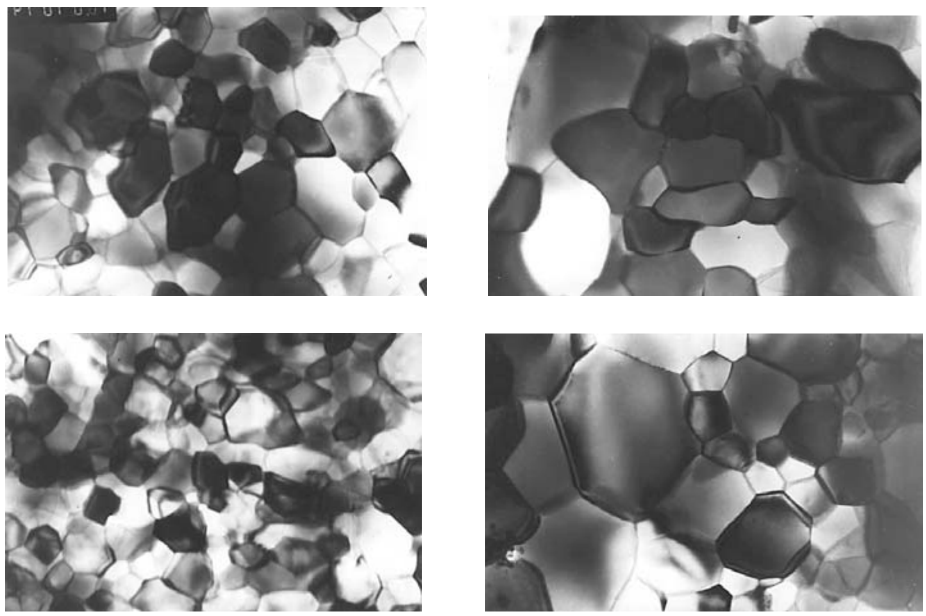

Figure 12. Microstructure of Nb_3Sn phase in bronze processed strands after heat treatment at 575 ^{o}C 150h + 650 ^{o}C 200h: a,b – diameter 0.81 mm; c,d – diameter 0.6 mm. (magnification 150 000).

The TEM analysis is local but in general the grain structure consists of the regions with relatively uniform sized small equaxed grains. Some regions could be found with much larger but also almost equal grains. These observations were valid for all investigated samples with slight tendency for diminishing of the average grain sizes with diminishing of the diameters of Nb3Sn filaments. A tendency for narrowing of the distribution of grain sizes was also found (Fig.13).

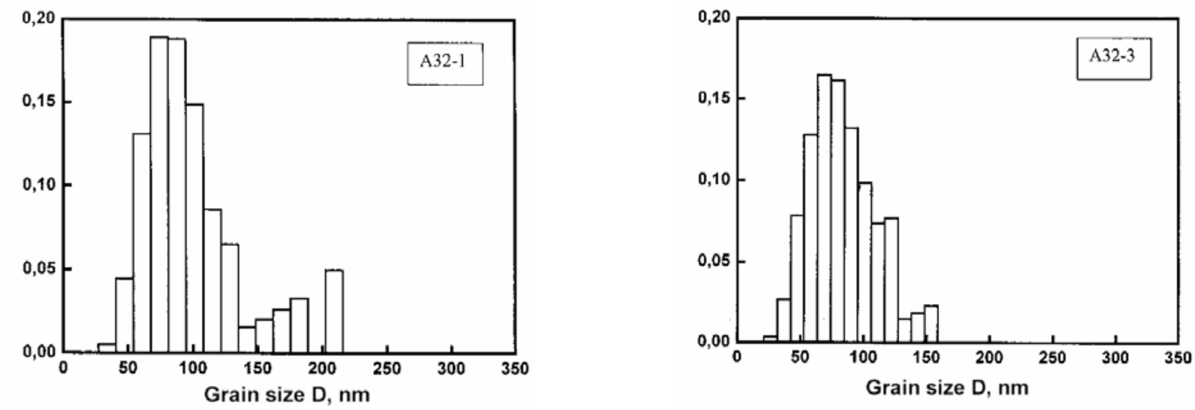

Figure 13. Distribution of the Nb3Sn grain size in bronze processed strands (a - 0.81 mm in dia and b - 0.6 mm in dia) after heat treatment at 575^0C 150h + 650^0C 200h [28].

Analyzing the data presented in the Fig.13 it could be stated that a further significant enhancement of the current carrying capability of the bronze processed wires could be attained by narrowing the distribution of grain sizes in the Nb3Sn layers, which corresponds in the plots to shifting the maximum position to the smaller size of grain.

In large magnet systems superconducting wires are usually used in the form of cables having different designs. In some cases (for example CICC) individual wires could be subjected to rather high mechanical loads, which are the combination of the thermal and electromagnetic forces. This is why the mechanical properties of Nb_3Sn wires became to be of primary importance. In BI several Nb3Sn wires with enhanced mechanical strength have been developed by replacing of the part of stabilizing Cu by high strength high conductivity microcomposite Cu-Nb material [29, 30]. The cross section of a strengthened Nb_3Sn wire is shown in the Fig.14.

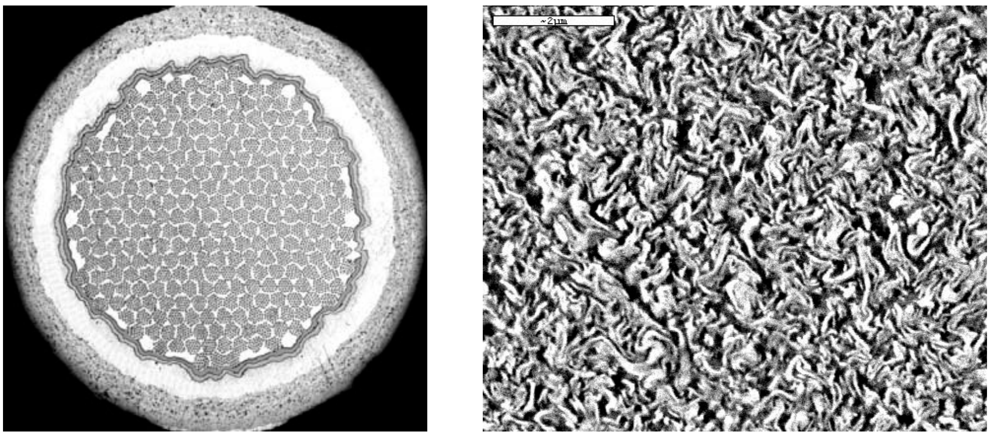

Figure 14. Cross section of strengthened Nb_3Sn wire –(a) and Cu-Nb strengthening material - (b)

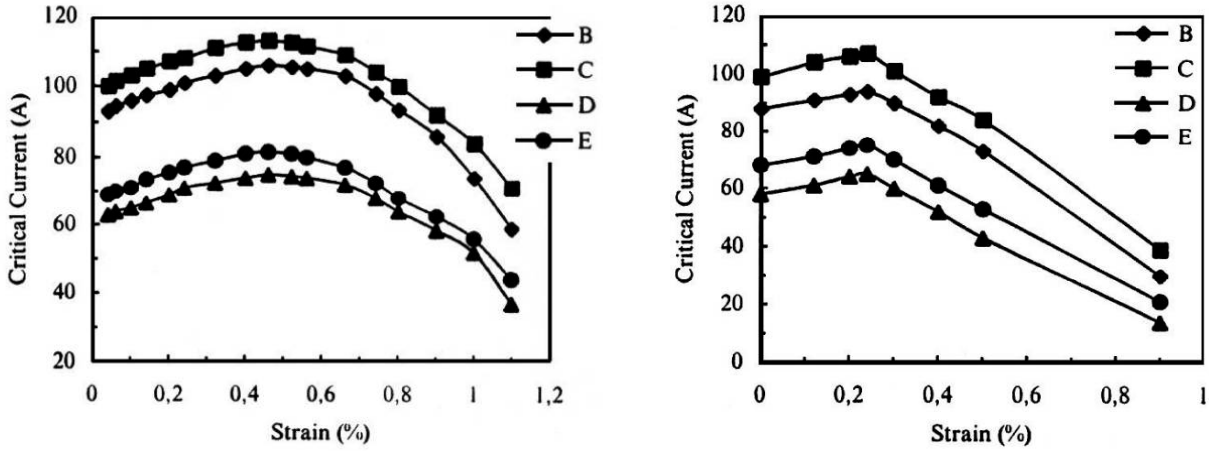

Figure 15. The influence of tensile strain on critical current in 12 T (B,C) and 14 T (D,E) 0.1 μV/cm (B,D) 1 μV/cm (C,E) in strengthened wire – (a) and without strenghening addition – (b).

It was shown that the ultimate tensile strength of the wire could be effectively increased up to 1000MPa in non-reacted state and up to 350 MPa after the reaction heat treatment. Critical current density of reinforced bronze processed wires remains essentially the same. The dependence of critical current density on tensile strain is shown in the Fig.15 [29].

3.2 Nb_3Sn wires – produced by internal tin method

In Nb_3Sn wires produced by internal tin method Nb filaments are embedded into a pure Cu matrix and Sn is provided in a form of separate sources distributed over the strand cross section: pure Sn or alloyed Sn may be used for the Sn sources. The larger Sn inventory available in the matrix of internal Sn strands (>20wt.%) allows a “reaction” of larger amounts of Nb, i.e. the achievement of a larger non-Copper critical current density.

On the other hand, the spacing between the filaments is much smaller, with the risk of frequent superconducting "bridges" during the Nb_3Sn formation, when the volume of the Nb filaments increases to ~ 33% in the area inside the diffusion barrier. These bridges build larger loops for the steady state magnetization currents in the filaments, resulting in large hysteresis losses. It was shown that hysteresis losses increased dramatically when distances between Nb_3Sn filaments decreased to less than 1 μm. Typical internal tin wires designed in BI are presented in Fig. 16.

In ITER type wires the combination of critical current density in the range of 800-1000 A/mm^2 and hysteresis losses lower than 1000 mJ/cm^3 are required. That is why in ITER type internal tin wires the volume fraction of the Nb_3Sn phase was only slightly increased in comparison to bronze processed wires. In high Jc type wires the limitation on the hysteresis losses is not applied, enabling to increase the volume fraction of Nb_3Sn phase significantly.

Nevertheless the design of the strands shown in Fig.16 have been developed in such a way that the amount of Nb_3Sn phase did not differ significantly (in case of ITER type strand the calculated volume fraction of Nb_3Sn phase was only 15% less than in High J_c strand), but the relations of the diameters of Nb filaments and spacings were significantly different. In ITER type strand the ratio d_f/Δ is 2.7 and in High Jc wires this ratio is 4.2. The absolute value of the spacing among filaments chosen was 2.5 μm for ITER type wires and and 1.1 μm for High J_c wires, which results in a high probability of the absence of bridging in ITER type strands and, on the contrary, high level of bridging in High Jc strands. Samples of both strands were subjected to the standard ITER heat treatment, consisting in the sequence: 575^0C 150h + 650^0C 200h. The superconducting properties are presented in table 1.

The grain structure of the samples investigated was generally similar to the grain structure of bronze processed strands described above. The following difference should however be noticed. The average Nb_3Sn grain size in internal tin strands was slightly larger than in bronze processed ones and the distribution of the grain sizes was generally wider [28]. It should be noted that insignificant differences in microstructures of both investigated internal tin strands could not explain the dramatic difference in their critical current densities (table 1) [31]. It was supposed that strong bridging in High J_c strand was the main reason of high critical current density through better redistribution of superconducting currents between filaments. At the same time strong bridging is negative for stability of the wires.

Due to larger volume fraction of the brittle intermetallic Nb_3Sn phase in internal tin wires the mechanical properties are even more important than for bronze processed wires. It should be also stressed that not only larger amount of brittle phase in the wire could lead to a possible faster degradation of critical current under applied strain, but also the presence of voids in the filaments region of the wire, that are consequence of reaction heat treatment accompanied by liquid phase formation and formation of intermediate Cu-Sn phases with different specific densities, could have a deteriorating effect. This is why the development of internal tin Nb_3Sn wires with enhanced mechanical strength was carried out in Bochvar Institute [32]. Two possible ways of introduction of strengthening microcomposite Cu-Nb elements in the design of final composite billet were tried. In Fig.17 the cross sections of both options are

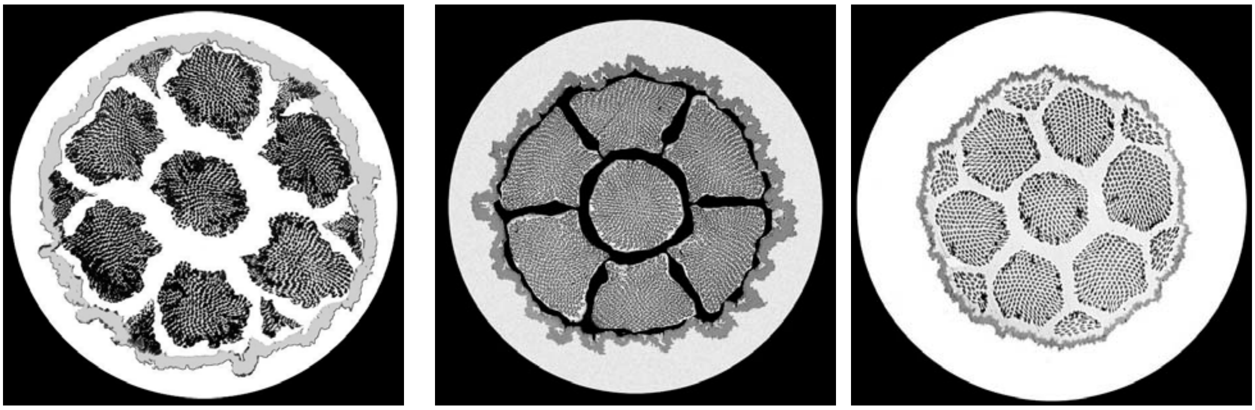

Figure 16. Typical internal tin wires designed in BI. High Jc wires – a),b); ITER type wire – c)

Table 1. Internal Tin Nb_3Sn wires superconducting properties

Definition	ITER type (Sn-P)	High Jc type (S12)
J_{nc}, non-Cu [A/ mm^2] @12 T, 4.2 K, 0.1 μV/cm, no external strain applied	745	2070
n value @12 T, 4.2K, between 0.1 to 1 μV/cm	22	16
B_{c2} (Kramer extrapolation) [T]	29.3	26.4
Tc, [K]	17.5	17.0
W_h, Hysteresis Loss [mJ/cm^3 non-Cu], @± 3T	270	>1000
Calculated Jc_{Nb3Sn} [A/ mm^2] @12 T, 4.2 K	2180	4850

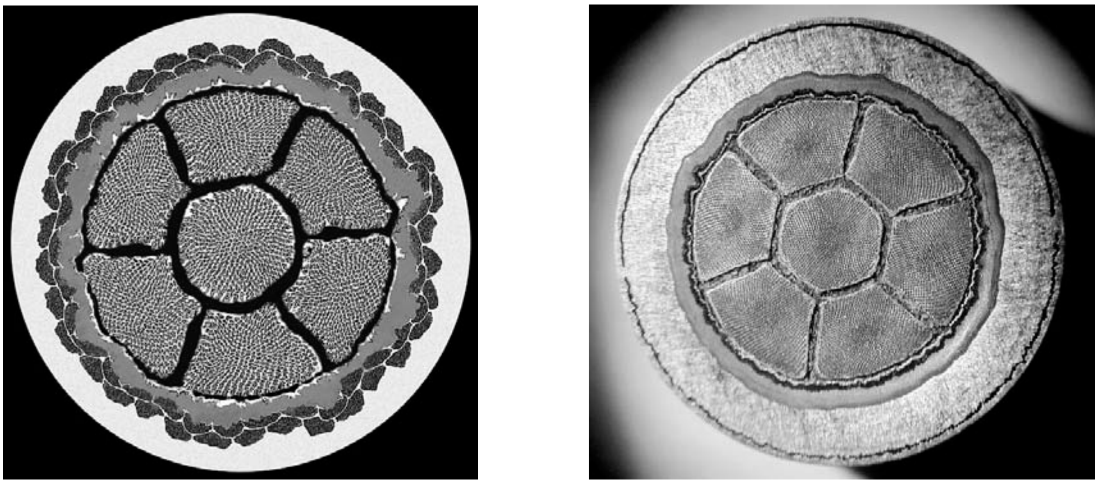

Figure17. Cross sections of internal tin Nb_3Sn wires strengthened by microcomposite Cu-Nb material in the form of rods (a) and tube (b).

shown with the arrangement of Cu-Nb material in the form of rods (fig.17a) and in the form of tube (fig.17b). The critical current density of the strengthened internal tin wire shown in Fig.17(a) was slightly higher than that of a wire with similar design but without reinforcement,, and attained 2376 A/mm^2 (12 T, 4.2 T) for a wire with 0.7 mm diameter.

Comparison of the mechanical properties of the strands with Cu-Nb reinforcing elements and without them before reaction heat treatment and after reaction heat treatment is presented in Fig.18. It was shown that the UTS values of strengthened internal tin Nb_3Sn wires were approximately 30% higher.

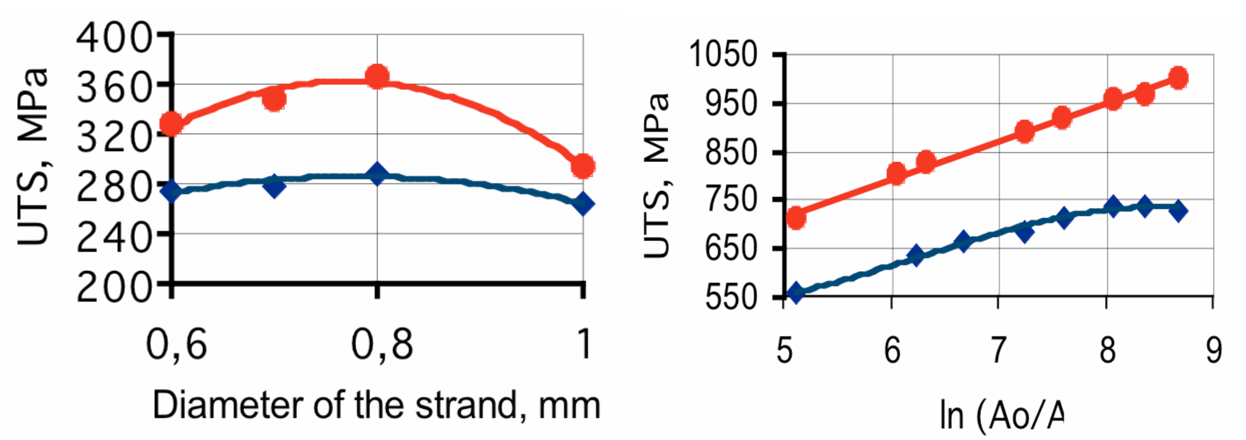

Figure 18. Mechanical properties of the internal tin wires with Cu-Nb reinforcing elements and without them before reaction heat treatment (Fig.18a) and after reaction heat treatment (Fig.18b)

4. SUMMARY

The limit of accelerator magnets wound from binary NbTi conductors and operated at 1.9K is in the range of 10 to 10.5T. We expect that this limit can be shifted to ~12T by the use of ternary alloy NbTiTa.

Nb3Sn wires seems to be most probable candidate materials for the application in both types of large future devices – fusion reactors and next generation accelerators, with magnet systems reaching 16 T.

Bronze processed Nb3Sn wires recently have made a good progress in critical current density, attaining 800 A/mm^2 (non-Cu, 12T, 4.2K) in commercially produced wires. In laboratory scaled wires 900 A/mm^2 (non-Cu, 12T, 4.2K) has been attained. Further increase of critical current density up to, potentially, 20% could be expected in bronze processed wires through the optimization of the strands designs and Nb3Sn layers microstructure.

Internal tin Nb3Sn wires with critical current density higher than 2000A/mm^2 (non-Cu, 12T, 4.2K) has been designed and fabricated. The question of stability of high Jc internal tin wires has to be investigated due to strong bridging of the filaments.

Mechanical strength of bronze processed and internal tin Nb3Sn wires could be effectively increased (up to 30-50%) by Cu-Nb microcomposite inserts.

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