Investigation on technical superconductors for large magnet systems in Bochvar Institute

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Introduction

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- Nb₃Sn strands - design and properties
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Introduction

- The progress in High Energy Physics and Fusion is relied on the successful realization of the currently existing Projects such as LHC and ITER. Magnet systems of both facilities consume around 1000 tons of superconductors each. Therefore the question of cost is one of the important factors.

- 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. Practically the choice of low temperature superconductors is limited to Nb$_3$Sn and Nb$_3$Al wires.
The possible superconductor candidates for high field magnetic systems are YBCO, Bi2212, Nb$_3$Al, Nb$_3$Sn.
The main objectives of R&D were to develop NbTi multifilamentary strands 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 formation.
NbTi strands – design and properties

• The nature of deformation of BCC NbTi filaments in FCC Cu matrix by 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. Taking into account that 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 the uniform deformation is substituted by the plane deformation and instead of cylindrical filaments the ribbon like filaments are formed.

To mitigate this effect the local volume fraction of NbTi should be properly chosen.

The major problems were resolved in the late 70-s and reliable technical NbTi strands with high critical current became commercially available. In 1978 the first tokamak T-7 with superconducting magnetic system has been successfully built and tested.

For Russian accelerator program UNK – 120 tons of NbTi strands with 8910 filaments ($6 \mu$m in dia) have been produced.

The critical current density up to 3000 A/mm² (5T, 4.2K) in commercially produced wires has been attained.
The limit of accelerator magnets wound from binary NbTi conductors and operated in superfluid helium at 1.9 K lied in the 10-to-10.5T range.

It is possible to raise $B_{c2}$ of NbTi by addition of Ta.

Two 4620 filamentary strands were designed and fabricated on the base of ternary NbTiTa alloys with 12 and 20 wt.% Ta (which layout meets the LHC specifications).

The main efforts were focused on the preparation of high homogeneous alloys and the optimization of heat treatments to achieve high $J_c$ at higher fields.

At 2K max $J_c$’s for NbTi-20wt%Ta strand reached 1550 A/mm² at 10 T and 750 A/mm² at 12 T.

At 1.85K $J_c$’s increased by 20% attaining 450 A/mm² at 13 T which was about twice higher than that for NbTi strands.
In the framework of ITER Project the RF Participant Team has manufactured the NbTi Cable (~0.5 ton), and shipped it to EFDA for further fabrication of PFCI. The testing of PFCI planned to be carried out in Japan in 2004.

Wire diameter – 0.73 mm
Number of filaments – 2346
Filament diameter – 9.8 mcm
Cu/non Cu Ratio - 1.4
Critical current density (by specification)

> 2700 A/mm² (5T, 4.2K)
(2800-2900 A/mm² – measured values)
VNIINM characterized the strands for PFCI in a wide range of temperature and magnetic fields. Comparison of CEA results with VNIINM ones was done for the five values of B explored.

For the major part of the data compared a satisfactory consistence between CEA and VNIINM 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 calls for differences in the structure of the tested strands (filaments structure, impurities ratio...).
NbTi strands – design and properties

NbTi strands designed for PF coils of ITER and extruded from 250mm in dia composite billets (Jc>2900A/mm² Filament diameter – 6 mcm)
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. The wire was fabricated by a single stacking method. Each filament (3.46 µm in dia) was surrounded by a matrix of 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 are inserted into hexagonal copper tube made from high purity Cu. The filament zone is 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 are fabricated from copper with \( R^{273}/R^{10} > 250. \)

The critical current density was > 2900 A/mm² (5T, 4.2K). The low hysteresis losses values of 51 kJ/m³ per wire and 144 kJ/m³ per superconductor volume have been achieved.
Nb$_3$Sn strands – design and properties

Two well established viable technologies are generally used for commercial production of Nb$_3$Sn strands – 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$_3$Sn 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 ($B_{c2}$).
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 is two phase material with brittle inclusions of intermetallic Cu-Sn phase.
The residual content of tin in bronze matrix after reaction heat treatment could be less than 0.5 %. Assuming stoichiometric composition of Nb$_3$Sn phase, the 25-26 % of the area inside the diffusion barrier should be occupied by Nb filaments.

**The maximum attainable volume fraction of Nb$_3$Sn 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$_3$Sn).

(or close to 30% in non copper region, including diffusion barrier).

Therefore the requirement of $J_{nc} > 800$ A/mm$^2$ (standard 12 T, 4.2K) assumes the attaining of critical current density in Nb$_3$Sn phase higher than 2670 A/mm$^2$, which is not a trivial task.
Disadvantage: 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.

Advantage: Extrusion of large composite billet is possible at the initial stage of deformation. Good metallurgical bonding of all elements of composite billet is guaranteed.
For Tokamak-15, a circulation-type conductor (340 m long, 17.4 mm by 6.5 mm in cross section) consisted of a Nb$_3$Sn-based superconducting transposed wire with 11 individual copper 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. The critical current of such a current-carrying element was not less than 8.5 kA in a field of 8 T. Approximately 90 t of current carrying elements were produced in an industrial way, which assumed production of more than 25 tons of strands.
In the frame of ITER model coils program VNIINM have designed and produced the strands for TF conductor coil-insert, which met the HP-2 specifications.

### Parameters and characteristics

- **Volume fraction of Cu** – 60%
- **Diameter of the strand** – 0.81 mm
- **Number of filaments** – 7225
- **Diameter of Nb<sub>3</sub>Sn filaments** – 2.5 mcm
- **Critical current density J<sub>c</sub>** (12 T; non-Cu) – > 550 A/mm<sup>2</sup>
- **Hysteresis losses Q<sub>h</sub>** (non-Cu; ±3T) – < 200 mJ/cm<sup>3</sup>
Bronze processed Nb$_3$Sn strands

Bronze processed wire with enhanced critical current properties has been developed in Bochvar Institute.

Jc(12T) = 774 A/mm$^2$
Hysteresis losses (+/-3T) = 337 mJ/cm$^3$
Cu/(non Cu) = 1.2
Quantity of filaments = 7851
bronze processed $\text{Nb}_3\text{Sn}$ strands

The cross section of the bronze processed strand with 12684 filaments

Scheme of the filaments design

The microstructure of $\text{Nb}_3\text{Sn}$ layers
Bronze processed \( \text{Nb}_3\text{Sn} \) strands could be produced with critical current density attaining 800 – 900 A/mm\(^2\).
Bronze processed $\text{Nb}_3\text{Sn}$ strands

Microstructure of $\text{Nb}_3\text{Sn}$ phase in bronze processed strands after heat treatment at $575^0\text{C}$ 150h + $650^0\text{C}$ 200h: a,b – diameter 0.81 mm; c,d – diameter 0.6 mm. (magnification 150 000).

22.03.2004
WAMS-2004 Archamps
bronze processed $\text{Nb}_3\text{Sn}$ strands

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 equaxed 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 $\text{Nb}_3\text{Sn}$ filaments. The tendency for narrowing of the distribution of grain sizes was also revealed.

Distribution of the $\text{Nb}_3\text{Sn}$ grain size in bronze processed strands (a - 0.81 mm in dia and b - 0.6 mm in dia) after heat treatment at $575^0\text{C}$ 150h + $650^0\text{C}$ 200h.
bronze processed **Nb$_3$Sn** strands

Critical current density of reinforced bronze wires rises from **735 A/mm$^2$** to **855 A/mm$^2$** (12T, 4.2K) with reduction in diameter of reinforced conductor from 0.8 mm to 0.5 mm.
The influence of tensile strain on critical current in
12 T (B,C) and 14 T (D,E)
0.1 mcV/cm (B,D)
1 mcV/cm (C,E)
The Nb filaments are imbedded into a pure Cu matrix and the Sn is provided in form of separate sources distributed over the strand cross section: pure Sn or alloyed Sn may be used for the 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 must decrease, with the risk of frequent superconducting "bridges" during the Nb$_3$Sn 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$_3$Sn filaments decreased to less than approximately 1 µm.
In ITER type strands the combination of critical current density in the range of 800-1000 A/mm² and hysteresis losses lower than 1000 mJ/cm³ are required.

In high Jc type strands the limitation on the hysteresis losses is not applied enabling to increase the volume fraction of Nb3Sn phase in the non copper area of the strand.
internal tin $\text{Nb}_3\text{Sn}$ strands

<table>
<thead>
<tr>
<th>Definition</th>
<th>ITER type</th>
<th>High Jc type</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strand diameter, mm</td>
<td>0.81</td>
<td>0.6</td>
</tr>
<tr>
<td>Twist pitch, mm</td>
<td>10</td>
<td>7</td>
</tr>
<tr>
<td>Volume fraction of stabilizing Cu, %</td>
<td>57</td>
<td>27</td>
</tr>
<tr>
<td>Barrier material</td>
<td>Ta</td>
<td>Ta</td>
</tr>
<tr>
<td>Number of Nb(Ti) filaments</td>
<td>1699</td>
<td>3673</td>
</tr>
<tr>
<td>Filament diameter ($d_f$) before reaction, µm</td>
<td>6.8</td>
<td>4.6</td>
</tr>
<tr>
<td>Filament spacing ($\Delta$), µm</td>
<td>2.5</td>
<td>1.1</td>
</tr>
<tr>
<td>Volume fraction of Nb inside Ta barrier, %</td>
<td>28.4</td>
<td>33.4</td>
</tr>
<tr>
<td>Volume fraction of Sn inside Ta barrier, %</td>
<td>16.4</td>
<td>19.8</td>
</tr>
<tr>
<td>Calculated volume fraction of $\text{Nb}_3\text{Sn}$ inside diffusion barrier, %</td>
<td>38.9</td>
<td>45.8</td>
</tr>
</tbody>
</table>
Cross sections of the internal tin strands after initial (homogenizing) stage of reaction heat treatment.

Much higher concentration of tin could be observed in high Jc wire. The voids are seen in the filaments area. Bridging of filaments is strong.
Microstructure of Nb$_3$Sn phase in internal tin High J$_c$ type strand after heat treatment with last stage at 575$^\circ$C 150h + 650$^\circ$C 200h (average grain size is approximately 90 nm).

The quality of Nb$_3$Sn phase is essentially the same as it was in bronze processed strands (uniaxed grains with rather significant distribution in sizes).
internal tin $\text{Nb}_3\text{Sn}$ strands

<table>
<thead>
<tr>
<th>Definition</th>
<th>ITER type</th>
<th>High Jc type</th>
</tr>
</thead>
<tbody>
<tr>
<td>$J_{nc}$, non-Cu critical current density [A/mm$^2$] @12 T, 4.2 K, 0.1 $\mu$V/cm, no external strain</td>
<td>745</td>
<td>2070</td>
</tr>
<tr>
<td>$n$ value @12 T, 4.2K, between 0.1 to 1 $\mu$V/cm</td>
<td>22</td>
<td>16</td>
</tr>
<tr>
<td>$B_{c2}$ (Kramer extrapolation) [T]</td>
<td>29.3</td>
<td>26.4</td>
</tr>
<tr>
<td>$T_c$, [K]</td>
<td>17.5</td>
<td>17.0</td>
</tr>
<tr>
<td>$W_h$, Hysteresis Loss [mJ/cm$^3$ non-Cu], @±3T</td>
<td>270</td>
<td>&gt;1000</td>
</tr>
<tr>
<td>Calculated $J_c$ (Nb3Sn) [A/mm$^2$] @12 T, 4.2 K</td>
<td>2180</td>
<td>4850</td>
</tr>
</tbody>
</table>

The results of the microstructure investigations enable to suggest that abrupt increase of $J_c$ (no proportional to the increase of volume fraction of Nb3Sn phase) is probably caused by strong bridging of filaments. At the same time strong bridging is negative for stability of the wires.
Internal tin Nb₃Sn strands

<table>
<thead>
<tr>
<th>Definition of the strand</th>
<th>Usual</th>
<th>Strengthened by Cu-18 %Nb alloy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter of the strand, mm</td>
<td>0,7</td>
<td>0,7</td>
</tr>
<tr>
<td>Filaments number</td>
<td>2947</td>
<td>2947</td>
</tr>
<tr>
<td>Volume fraction of stabilization, %</td>
<td>38</td>
<td>40</td>
</tr>
<tr>
<td>Critical current density (non Cu), (12 T, 4.2 K), A/mm²</td>
<td>2273</td>
<td>2376</td>
</tr>
<tr>
<td>Hysteresis losses (±3 T, 4.2 K), mJ/cm³</td>
<td>~1000</td>
<td>~1200</td>
</tr>
</tbody>
</table>
internal tin $\text{Nb}_3\text{Sn}$ strands

Comparison of the mechanical properties of the strands with Cu-Nb reinforcing elements and without them before reaction heat treatment (upper graph) and after reaction heat treatment (lower graph)

The UTS values of strengthened internal tin wires are approximately 30% higher.
High field properties of ITER-type $\text{Nb}_3\text{Sn}$ strands

Bronze processed wires

Internal tin wires

(Measured in Grenoble High Magnetic Field Laboratory)
Summary

The limit of accelerator magnets wound from binary NbTi conductors and operated at 1.9K equal to 10-10.5T can be shifted to ~12T by the use of ternary alloy NbTiTa.

Nb$_3$Sn 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 Nb$_3$Sn 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 20% potentially could be expected in bronze processed wires through the optimization of the strands designs and Nb$_3$Sn layers microstructure.

Internal tin Nb$_3$Sn 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 Nb$_3$Sn wires could be effectively increased (up to 30-50%) by Cu-Nb microcomposite inserts.