# Powder-in-Tube (PIT) $Nb_3Sn$ Conductors for High-field Magnets

J.L.H. Lindenhovius and E.M. Hornsveld
ShapeMetal Innovation B.V., Enschede, The Netherlands

A. den Ouden, W.A.J. Wessel and H.H.J. ten Kate
University of Twente, Enschede, The Netherlands

*Abstract*--**New $Nb_3Sn$ conductors, based on the powder-in-tube (PIT) process, have been developed for application in accelerator magnets and high-field solenoids. For application in accelerator magnets, SMI has developed a binary 504 filament PIT conductor by optimizing the manufacturing process and adjustment of the conductor lay-out. It uniquely combines a non-copper current density of 2680 A/mm$^2$@10 T with an effective filament diameter of about 20 μm. This binary conductor may be used in a 10 T, wide bore model separator dipole magnet for the LHC, which is being developed by a collaboration of the University of Twente and CERN. A ternary (Nb/7.5wt%Ta)$_3$Sn conductor containing 37 filaments is particularly suited for application in extremely high-field superconducting solenoids. This wire features a copper content of 43 %, a non-copper current density of 217 A/mm$^2$ @ 20 T and a $B_{c2}$ of 25.6 T. The main issues and the experimental results of the development program of PIT $Nb_3Sn$ conductors are presented and discussed in this paper.**

## I. Introduction

The strongly improved properties of the brittle $Nb_3Sn$ conductors [1], [2] and their availability on industrial scale make them specifically attractive for application in demanding magnet systems like high-field solenoids and accelerator magnets. Whereas the emphasis for the first application lies on a high $B_{c2}$ and a high $J_c$ at about 20 T, the demands for the latter also include a small filament diameter and a well-controlled copper fraction. Powder-in-tube (PIT) processed $Nb_3Sn$ conductors have proven to be a good candidate for both applications.

The PIT method for manufacturing superconducting $Nb_3Sn$ wire is characterized by niobium tubes containing a powder of the inter-metallic compound $NbSn_2$. Usually after coil winding a relatively short heat treatment of 40-100 hours at 675°C is needed for the formation of the superconducting $Nb_3Sn$ phase. By diffusion, the tin from the powder core reacts with the niobium and a $Nb_3Sn$ layer grows until about 2/3 of the niobium wall has reacted. The excess of niobium tube material outside the $Nb_3Sn$ layer acts as a natural barrier to avoid contamination of the copper matrix. Since the tin is confined in the tubes and the filaments are directly arranged in the copper matrix, this process should in principle enable a further reduction of filament size with perfectly de-coupled filaments.

The precursor material for the powder core, the niobium tubes and the matrix can be manipulated more or less independently within limits imposed by mechanical rigidity and billet processing. Therefor the PIT-process appears to be particularly suited for incorporation of (artificial) pinning centers in the $Nb_3Sn$ layer by local additions [3],[4].

A first step into this direction is the development of a 37 filament high-field conductor utilizing Nb/7.5wt.%Ta tubes, which should particularly increase the $B_{c2}$ and the $J_c$ in the 20 T range.

At the same time SMI is continuously improving the properties of the binary PIT-$Nb_3Sn$ conductors for application in accelerator magnets. Both programs are discussed in this paper.

## II. Binary PIT Conductor for Accelerator Magnets

### A. Development Program

The lay-out and corresponding operating parameters for any next generation accelerator (VLHC, muon collider etc.) are still under discussion. Nevertheless, general requirements for any superconductor to be used in this field may be summarized as follows: a strain insensitive, cheap, easy-to-cable conductor exhibiting an unprecedented $J_c$ in the field range of 10-15 T, filaments smaller than 20 μm and an adequate copper fraction for reasons of stability and protection. Conductor development programs should aim at improvements on all aspects at the same time.

In this perspective SMI has decided as a first step to reduce the effective filament size in the PIT conductors while maintaining a non-copper $J_c$ > 2200 A/mm$^2$ @ 10 T with a copper fraction close to 50 %. With the manufacturing of a

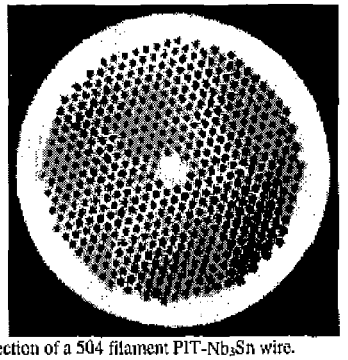

Fig. 1. Cross section of a 504 filament PIT-$Nb_3Sn$ wire.

Manuscript received September 27, 1999

1051-8223/00$10.00 

TABLE I
CHARACTERISTICS OF THE INVESTIGATED PIT-$Nb_3Sn$ WIRES

| filaments | 192 | 492 | 504 |
|---|---|---|---|
| manufacturer | ECN | SMI | SMI |
| diameter (mm) | 0.90 | 0.91 | 0.90 |
| copper fraction (%) | 52 | 54 | 52 |
| billet reduction | cold drawn | cold drawn | extruded and cold drawn |

wire containing 492 filaments showing an effective filament diameter close to 20 μm, a first encouraging result has been achieved [2]. However, this wire showed a relatively low $J_c$ of 1900 A/mm$^2$ @ 10 T. Adjustment of the filament lay-out, the powder content and the manufacturing procedures has resulted in a 504 filament wire that uniquely meets most electro-magnetic demands. For the first time a cold hydrostatic extrusion has been applied successfully in the PIT-process to improve the internal bonding and to increase the yield. Fig. 1 shows the cross-section of this wire. Table I summarizes the main characteristics of both wires, together with the standard 192 filament wire, that performed so well in the 11 T dipole MSUT [5].

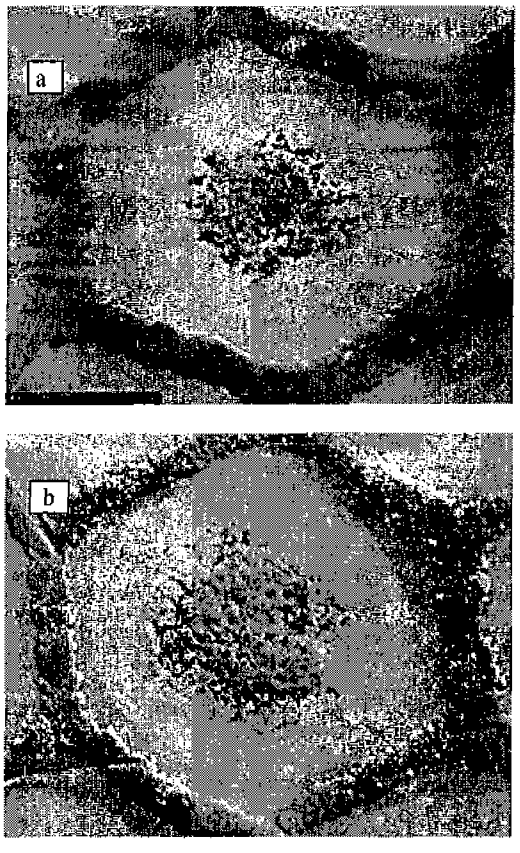


Fig. 2. SEM picture of the cross section of a single filament after the heat treament of a 492 filament (a) and a 504 filament wire (b).

SEM pictures of single reacted filaments from both the PIT-492 and the PIT-504 wire are presented in Fig. 2. While the wire lay-out differs only slightly, the reacted $Nb_3Sn$ region in the PIT-492 is much smaller than in the PIT-504 case. Optimizing the composition of the core precursors clearly results in a more efficient use of the available niobium tube. EDX scans reveal that after reaction no tin is left in the core of the PIT-492 filaments, while the PIT-504 cores still contain some tin, even after the optimal heat treatment.

### B. *Critical Current Measurements*

Critical current measurements are performed on 1 meter long samples at 4.2 K. The samples are mounted, heat treated and fixed on a standard ITER type of TiAlV sample holder. In all cases an $I_c$ criterion of 10 μV/m is applied. Note that due to the lower thermal expansion of TiAlV, the obtained values for $I_c$, $B_{c2}^*$ and $T_c$ are about 3 % higher than when a stainless steel sample holder is used.

Fig. 3 shows the obtained non-copper critical current density from PIT-492 samples and PIT-504 samples after different heat treatment times. As stated earlier, the $I_c$ of the PIT-492 is low due to a lack of tin in the core. The PIT-504 shows a very high $I_c$ corresponding to a current density of 2680 A/mm$^2$ @ 10 T in nicely de-coupled filaments after a heat treatment of only 47 hours at 675 $^0$C. Irrespective the heat treatment or the wire composition, the extrapolated Kramer $B_{c2}^*$ amounts to 21.2 +/ 0.1 T for all investigated samples.

The PIT-504 wire may be used in a wide bore, 10 T model dipole magnet that is currently developed by the University of Twente and CERN [6]. For this magnet a non-copper (50 %) current density of 1300 A/mm$^2$ @10.8 T is required, which is about 40 % lower than the measured

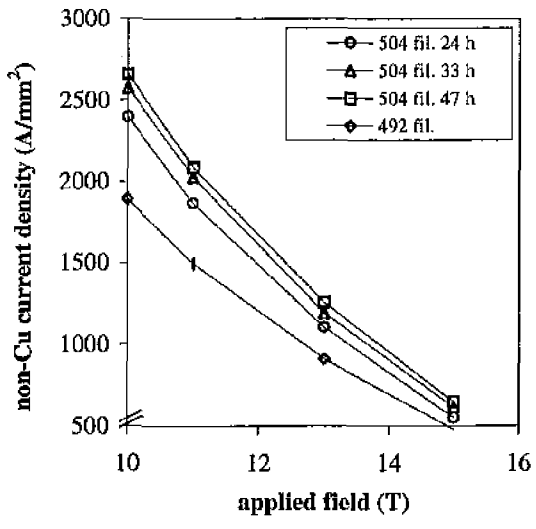


Fig. 3. Non-copper critical current density versus applied field of a binary 504 filament wire for different heat treatment times and a 492 filament wire.

value in this wires. First cabling experiments however reveal undesired filament damage after the cabling process and consequently a much higher $I_c$ degradation than the PIT-192 and the PIT-492 wires. Further investigations on this issue are being carried out [7].

### C. Magnetization Measurements

SEM analysis is helpful but not sufficient for the determination of the effective filament diameter. Therefor, the magnetization of the mentioned wires has been measured in a set-up using a double compensated pick-up coil set as part of a fully integrating flux-transformer circuit.

Above the penetration field $B_p$, the magnetic moment per unit volume in a superconducting wire containing hollow, tube-like filaments (Fig. 2) may be written as [8] :

$$M = \frac{2}{3\pi} \frac{n J_c D_{of}^3}{D_w^2} \left[ 1 - \left( \frac{D_{if}}{D_{of}} \right)^3 \right] . \qquad (1)$$

In equation (1), $n$ is the number of filaments, $J_c$ the critical current density in the $Nb_3Sn$ layer, $D_{of/if}$ is the outer/inner diameter of the $Nb_3Sn$ ring and $D_w$ the wire diameter. Up to fields of about 7 T, $J_c$ is adequately described by the Kim relation :

$$J_c(B) = \frac{J_0 B_0}{(B_0 + |B|)} . \qquad (2)$$

In equation (2) $B$ is the local field, $J_0$ and $B_0$ are the Kim parameters which are assumed to be constant for a specific $Nb_3Sn$ conductor.

TABLE IV
FIT PARAMETERS FOR THE KIM RELATION

| | 492 fil. | 504 fil. |
|---|---|---|
| inner $Nb_3Sn$ diameter (μm) | 10 | 12 |
| outer $Nb_3Sn$ diameter (μm) | 19 | 22 |
| $J_0$ (A/m$^2$) | $1.61 \times 10^{11}$ | |
| $B_0$ (T) | 0.605 | |

Fig. 4 shows the measured and calculated magnetization per unit wire volume of a PIT-492 and a PIT-504 sample. The fit parameters are listed in Table IV. The dimensions for the $Nb_3Sn$ region are derived from the SEM pictures. For both samples a reasonably good fit is obtained with the same Kim parameters. Due to lack of representative values for the Kim parameters, this fit still does not exclude filament bridging.

A strong additional check on the occurrence of filament bridging is the extension of the Kramer relation derived from high field $I_c$-measurements (B > 10 T) to lower fields. In the absence of filament bridging, the calculated $I_c$ derived from this top-down Kramer extrapolation on one hand and the Kim $I_c$ on the other hand should coincide below about 7 T. Fig. 5 shows the result of this extrapolation for the PIT-504 sample. The independently obtained critical currents agree very well for 1 < B < 7 T, which implies that the effective filament diameter equals the physical reacted $Nb_3Sn$ region.

The absence of bridging is a typical property of PIT-processed wires, in which the filaments are inherently decoupled as a result of the $Nb_3Sn$ formation inside the tin-shielding niobium tubes.

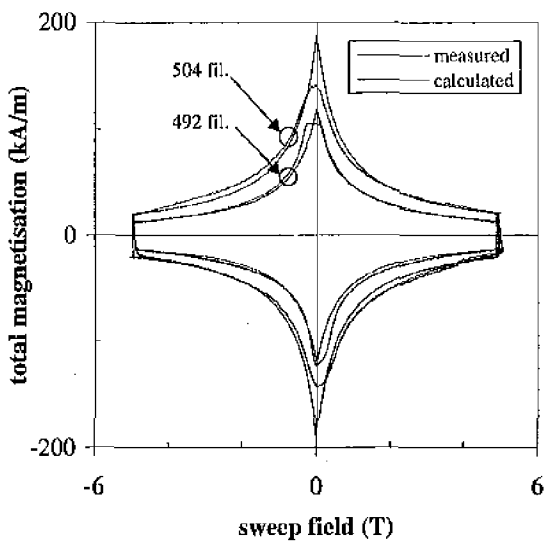


Fig. 4. Calculated and measured magnetization per unit volume of a PIT-492 and a PIT-504 sample. The sinusoidal field is swept with a frequency of 1 mHz.

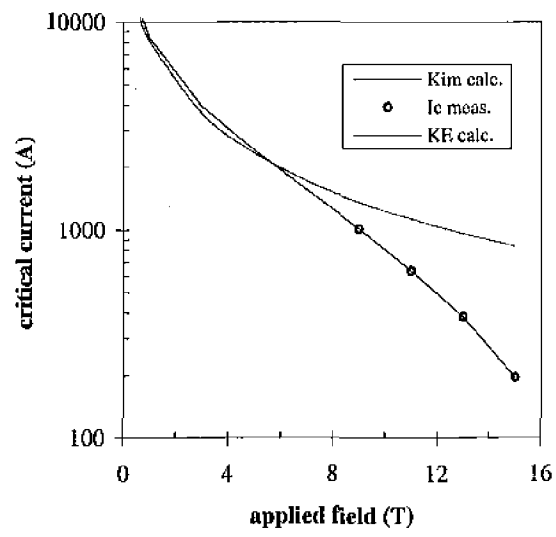


Fig. 5. Calculated critical current derived from magnetization measurements (Kim calc.) at low field and from direct $I_c$ measurements (KE calc.) at high field.

## III. Ternary PIT Conductors for High Fields >20 T

For the very first time SMI has manufactured a ternary $(Nb/7.5wt.\%Ta)_3Sn$ conductor with 37 filaments and a copper fraction of 43 % following the traditional procedures for stacking and wire drawing.

The critical current of two independently prepared samples of a 0.8 mm ternary wire has been measured. The first sample is mounted on a standard ITER type TiAlV sample holder for measurements in a background field of $10 < B_a < 15.5$ T (LFM). The second sample, mounted on a smaller diameter TiAlV holder, is measured in fields above 17 T (HFM).

For both samples the overall current density at 4.2 K is presented in Fig.6, together with the corresponding n-values. The applied $I_c$ criterion is 10 μV/m. Also plotted is a Kramer extrapolation of the LFM-measurements upwards which agrees very well with the HFM-results. The Kramer $B_{c2}^*$ is 25.5 T, while the actual measured $B_{c2}$ is 25.6 T. At 20 T, the non-copper $J_c$ (corrected for the copper current) amounts to 217 A/mm$^2$ with an n-value of 20. Compared to the 37-filament binary conductor with the same lay-out, the current density of the ternary PIT-$Nb_3Sn$ conductor becomes superior for fields above 11.5 T.

These very encouraging results after the manufacturing of a ternary $(NbTa)_3Sn$ conductor with a non-optimized standard lay-out show the potential of the PIT-processed superconductors in the very high-field range as well. A next step in this development program will be to investigate the effect of doping of the tube and powder precursor materials on the critical properties $B_{c2}$, $T_c$ and $J_c$.

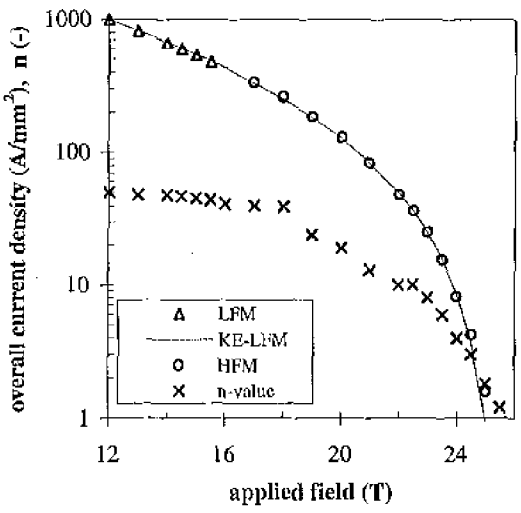


Fig. 6. Overall critical current density and n-values versus applied field for a $(Nb,7.5wt.\%Ta)_3Sn$ wire obtained from both low field (LFM) and high field (HFM) measurements. KE-LFM represents a Kramer extrapolation derived from the low-field measurements.

## IV. Conclusions

New $Nb_3Sn$ conductors, based on the powder-in-tube (PIT) process, have been developed. A ternary $(Nb/7.5wt\%Ta)_3Sn$ conductor containing 37 filaments is particularly suited to application in extremely high-field superconducting solenoids. This wire features a copper content of 40 %, a non-copper current density of 217 A/mm$^2$ @ 20 T and a $B_{c2}$ of 25.6 T. For application in accelerator magnets, SMI has developed a binary 504 filament PIT conductor by optimizing the manufacturing process and adjustment of the conductor lay-out. It uniquely combines a non-copper current density of 2680 A/mm$^2$@10 T with an effective filament diameter of about 20 μm. This binary conductor may be used in a 10 T, wide bore model separator dipole magnet for the LHC, which is being developed by a collaboration of the University of Twente and CERN.

## Acknowledgment

The critical current measurements at fields above 17 T have been carried out at the National High Magnetic Field Laboratory, Tallahassee, Florida, USA.

## References

[1] T. Pyon and E. Gregory, "Niobium-Tin conductors for fusion, high energy physics and other applications", *IEEE Transactions on Applied Superconductivity*, June 1999, vol. 9, number 2

[2] I.H. Lindenhovius, E.M. Hornsveld, A. den Ouden, W.A.J. Wessel and H.H.J. ten Kate, " Progress in the development of $Nb_3Sn$ conductors based on the 'Powder in Tube' metgod with finer filaments", *IEEE Transactions on Applied Superconductivity*, , vol. 9, number 2, June 1999

[3] D.R. Dietderich, M. Kelman, J.R. Litty and R.M. Scanlan, "High critical current densities in $Nb_3Sn$ films with engineered microstructures -- artificial pinning microstructures, " *Advances in Cryogenic Engineering, (Materials)* , vol. 44 (1998)

[4] K. Tachikawa, S. Yamamoto, T. Yokoyama and T. Kato, "New high-field $Nb_3Sn$ conductors prepared from Ta-Sn compound powder", *IEEE Transactions on Applied Superconductivity*, June 1999, vol. 9, number 2

[5] A. den Ouden et al, "Application of $Nb_3Sn$ superconductors in accelerator magnets", *IEEE Trans. on Applied Superconductivity*, vol. 7, 1997, p. 733

[6] A. den Ouden et al, " A 10 T model separator magnet for the LHC", *proceedings of the 15th International Conference on Magnet Technology*", Beijing, 1997

[7] A. den Ouden et al, "Development of a wide bore 10 T $Nb_3Sn$ model dipole magnet", to be presented at this conference

[8] J. Eikelboom, "*AC. Loss in prototype conductors for the NET toroidal field coils*", PhD. thesis, University of Twente, 1991