Journal of Physics: Conference Series



# Recent progress in a development of $Nb_3Sn$ internal tin strand for fusion application

To cite this article: S V Sudyev *et al* 2010 *J. Phys.: Conf. Ser.* **234** 022037

# Recent progress in a development of $Nb_3Sn$ internal tin strand for fusion application

**S V Sudyev[1], A K Shikov[1], V I Pantsyrny[1], A E Vorobieva[1], N A Beliakov[1], V V Sergeev[1], I A Golovchanskiy[2], S M Zernov[3] and K M Abramushin[4]**

[1]Bochvar Research Institute of Inorganic Materials (JSC «VNIINM»), 5a Rogova Street, 123098 Moscow, RF

[2]Moscow Engineering Physics Institute, 31 Kashirskoe shosse, 115409 Moscow, RF

[3]JSC «TVEL», 49 Kashirskoe shosse, 115409 Moscow, RF

[4]JSC «Chepetsky Mechanical Plant» (JSC CMP), 7 Belova Street, 427620 Glazov, RF

E-mail: soudiev@id.ru

**Abstract**. The layout optimization of ITER-type $Nb_3Sn$ internal tin (IT) superconducting strand for magnet systems of future fusion reactors was carried out aiming on the large scaled industrial production. The new approach to a formation of the initial composite billets for fabrication of IT superconducting strand has been proposed. This approach assumes the use of round shaped instead of hexagonal shaped Cu-Nb subelements covered by tin layers. The positive influence of the use of round shaped multifilamentary Cu-Nb subelements on the uniformity of filaments array in the final strands has been shown. The investigation on the use of composite filaments consisting of multiple niobium rods and rods made from NbTi alloy was carried out. It was shown that the use of proposed composite filaments enabled to effectively regulate the grain size of niobium in Cu-Nb subelements before the cold deformation by drawing. The experimental data on the influence of the proposed composite design of the filaments on the kinetics of $Nb_3Sn$ layer formation process are given. The relations of microstructure and properties of $Nb_3Sn$ IT strand with modified design have been analyzed. The superconducting properties attained met the main ITER requirements for the TF strands with non-copper critical current density higher than 800 A/mm$^2$ (4.2 K; 12 T) and hysteresis losses (±3 T) less than 500 mJ/cm$^3$.

## 1. Introduction

It is well known that IT $Nb_3Sn$ strands are characterized by higher critical current capacity compared to bronze processed wires [1]. Therefore the advanced fusion reactors magnet systems rely mainly on the IT $Nb_3Sn$ superconductors with relatively high critical current and for ITER project the IT $Nb_3Sn$ strands for toroidal field coils and for Central solenoid will be supplied by the most manufactures from the participants countries [2]. The application of IT superconductors having high critical current capacity is also very perspective for other large-scale magnet systems [3, 4]. The existing level of technology allows to create economically effective industrial production of IT $Nb_3Sn$ superconductors.

Nevertheless, during ITER R&D stage the certain specific problems were identified connected with the application of IT strands.

Model Coils program undertaken in the framework of ITER Project revealed a new effect of degradation of the strands in the scope of large cable-in-conduit conductor (CICC). This effect is associated with the intrinsically typical for IT strand occurrence of large areas of brittle intermetallic $Nb_3Sn$ phase within strand matrix. A lot of researchers performed their works in that area trying to minimize this effect of degradation. Main objective of this research was also to develop IT strand with controlled strand geometry that will eliminate the formation of crucial excessive bridging and, respectively, mitigate the effect of degradation.

In the production process of IT $Nb_3Sn$ superconductors for fusion application, in a strand layout in order to attain higher critical current densities in magnetic fields higher than 12 T the filaments doped by Ti or Ta are used. The metallurgical doping of Nb by Ti or Ta in principle allows to produce the high quality initial ingots with fine-grained structure. However the fabrication of such precise homogeneous initial ingots requires the use of rather complicated technological processes with manufacturing of initial electrodes and following electron beam melting of niobium alloys such as Nb-1.2wt.%Ti or Nb-7.5wt.%Ta. These alloys enable to obtain the $Nb_3Sn$ filaments of necessary quality in strands, but as a result the cost of industrial IT $Nb_3Sn$ strands for fusion application and another large-scale magnet systems goes up.

In this experimental work it was proposed to use artificial Ti doping of filaments formed from non-doped niobium rods altogether with the rods made from conventional Nb-47wt.%Ti alloy [5]. With the purpose of the formation of predetermined fine grain structure in Nb filaments they were formed from the set of Nb elements placed in a copper alloy matrix and which consisted of the separate Nb components subjected to significant initial deformation for substantial decreasing of Nb grain size.

With this composite artificially doped by titanium Nb filaments three experimental IT $Nb_3Sn$ strands were formed with a design that was supposed to formal meet the ITER requirements. Designed experimental superconducting IT strands were mainly differentiated one from another by the procedures of preparation of initial filaments billets artificially doped by Ti from rods of industrially produced alloy Nb-47wt.%Ti. The processes of hot extrusion of the billets consisted of the Nb core and tube, cold rolling and drawing for Nb rods were used.

This research and development work was carried out with the use of the new industrial equipment for ITER strands production at the plant established on JSC CMP in Glazov (RF) in cooperation with JSC “TVEL”, JSC “VNIINM” and State Atomic Energy Corporation “Rosatom”. Nowadays, at this plant the production of $Nb_3Sn$ strands for ITER Project by bronze technology is under the way altogether with the NbTi strands also for ITER Poloidal coils PF1 and PF6. Thus, one of the side objective of this work was the verification of the possibility to use the newly installed industrial equipment of JSC CMP for an industrial production of advanced IT $Nb_3Sn$ superconducting strands.

## 2. Experimental Procedure

The new approach to a formation of the initial composite billets for the fabrication of IT $Nb_3Sn$ superconducting strands briefly identified in the introduction has been proposed and is schematically presented at Figure 1.

The initial Nb ingot 250 mm in diameter with high homogeneous chemical composition and uniform grain structure was produced in JSC CMP. This ingot was cut into several pieces after hot extrusion process. From two Nb pieces Nb tube and Nb core were produced by hot extrusion process for further assembling of the composite billet presented at Figure 1a. From another Nb pieces two Nb sets of rods were produced by hot extrusion process for a formation of the hexagonal rods for assembling of the composite billets presented at Figure 1b and Figure 1c. The composite billet at Figure 1b consists of Nb hexagonal rods produced by cold rolling process and the composite billet at Figure 1c consists of Nb hexagonal rods produced by cold drawing process respectively.

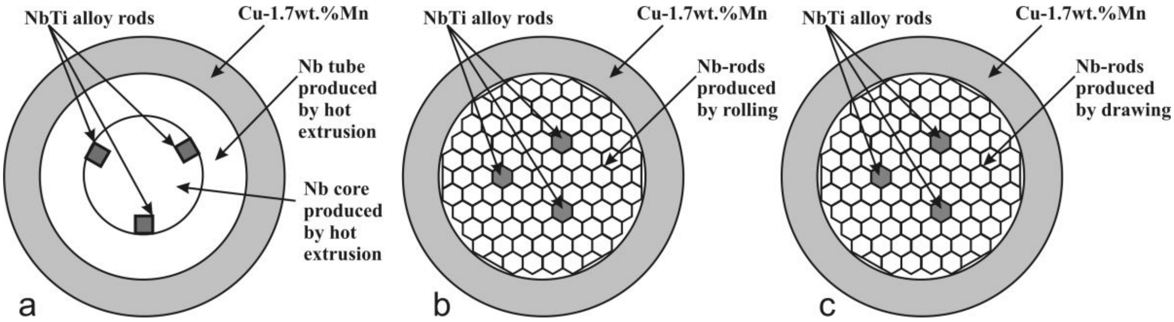



**Figure 1.** Formation of the initial composite billets for fabrication of composite filaments of advanced IT $Nb_3Sn$ superconducting strands: a – made from Nb tube and Nb core produced by hot extrusion; b – made from Nb rods produced by cold rolling process; c – made from Nb rods produced by cold drawing process.

The three NbTi alloy rods were also produced by hot extrusion process for incorporation in the composite billet presented at Figure 1a. The two sets of three NbTi alloy rods each to be placed into the composite billets shown at Figure 1b and Figure 1c, were produced by cold rolling and drawing processes respectively. All NbTi alloy rods were produced from one piece of initial Nb-47wt.%Ti alloy ingot.

The composite billets formation is presented schematically at Figure 2. The outer covering tubes for filament composite billets were produced from Cu-1.7wt.%Mn alloy. The positive influence of small manganese addition in a Cu matrix on the kinetics of $Nb_3Sn$ layer formation and on the reduction of hysteresis losses level was analyzed and confirmed in the earlier papers [6, 7]. The filament composite billets were hot extruded and cold drawed to predetermined size. These rods were used for forming of the multifilamentary subelements for final strands billets by new approach. This approach assumes the use of round shaped instead of hexagonal shaped multifilamentary subelements covered by tin layers. The scheme of this new design is presented at Figure 3. The positive influence of the use of round shaped multifilamentary subelements on the uniformity of filaments array in the final strands has been shown.

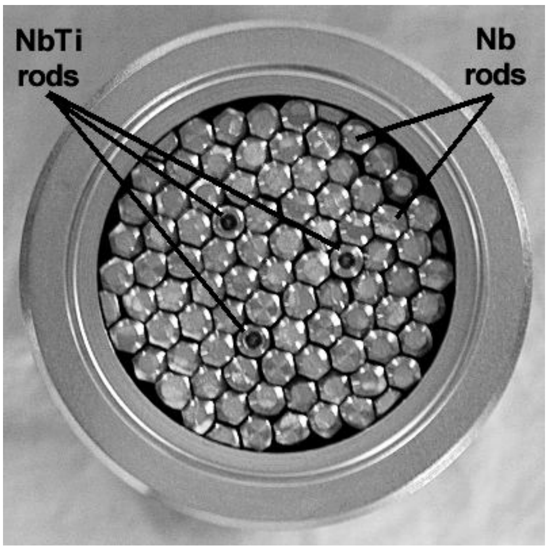



**Figure 2.** Filament composite billet design.

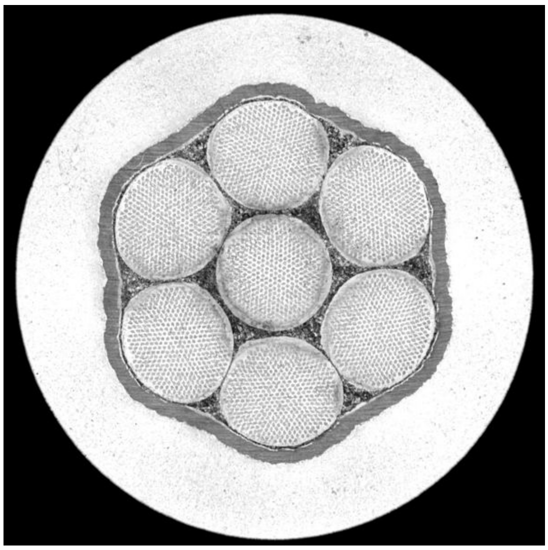


**Figure 3.** Final strand restack layout.

Three types of $Nb_3Sn$ multifilamentary strands with distributed source of tin have been fabricated by internal-tin method. The typical cross-section of these superconducting strands before reaction heat treatment (0.82 mm in dia) is presented in Figure 4. The parameters of the fabricated superconducting strands are given in Table 1. These composite wires consist of round multifilamentary tin-coated modules (subelements) that are inserted in Ta tube diffusion barrier with outer stabilizing copper. The round multifilamentary modules assumed to be used in the each designed superconducting strands contain 421 individual filaments. Their formation scheme and composite billet layouts presented at Figure 1 respectively.

The single filament diameter in superconducting strands was determined as a result of the analysis of digital images made by the scanning electron microscope, by a number of pixels forming respective areas of images of superconductors cross-sections.

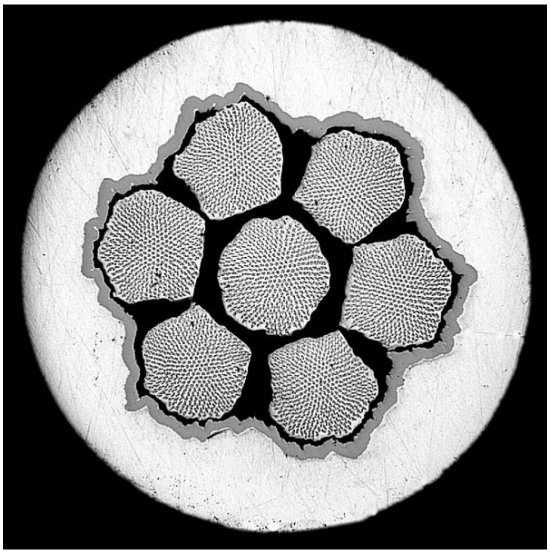


**Figure 4.** Typical cross-section of IT superconducting strand (0.82 mm in dia) formed from round multifilamentary subelements covered by tin layers.

**Table 1.** Parameters of the fabricated IT superconducting strands.

| Strand identification | ITS01-08 | ITS02-08 | ITS15-08 |
|---|---|---|---|
| Filament layout | Figure 1a | Figure 1b | Figure 1c |
| Outer diameter of the strand, mm | | 0.82 | |
| Volume fraction of Cu stabilization, vol.% | | 50 | |
| Filaments amount | | 2947 | |
| Strand twist pitch (right hand), mm | | 15 | |
| Diffusion barrier material | | Ta | |
| Nb/NbTi filament composition | | Nb-1.2wt.%Ti | |
| Matrix composition of Nb/NbTi filament | | Cu-1.7wt.%Mn | |
| Volume fraction of Nb filaments inside barrier, vol.% | | 32 | |
| Nb/NbTi filament diameter, μm | | 5.6 | |
| Spacing between Nb/NbTi filaments, μm | | 2.3 | |

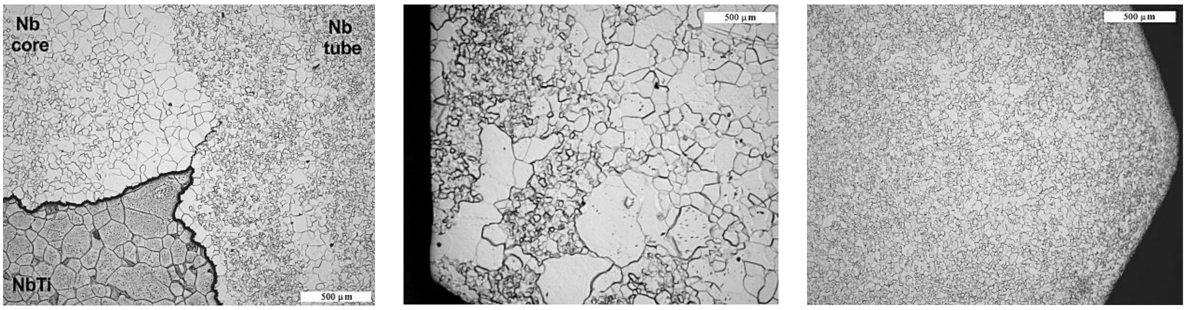
It should be noted that all developed superconducting wires have uniform parameters of the strand design and varies only by filament layouts, which presented at Figure 1 respectively.

## 3. Results and Discussion

### *3.1. Recrystallization processes and heat treatment*

It is clear that one of the key operation strongly influencing on the final uniformity of the filaments geometry is the operation of the recrystallization of the subelements before their assembling into the final billet. Different heat treatments for primary recrystallization of heavily deformed niobium components after different cold deformation routes were proposed and performed.

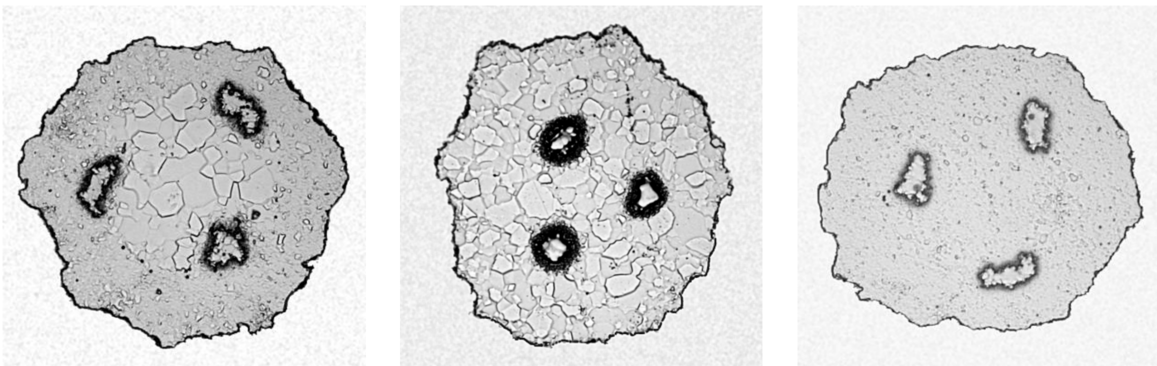
First recrystallization heat treatment was carried out on initial Nb elements from filament composite billets. The microstructure of the recrystallized Nb rod is presented at Figure 5. The applied heat treatment was the same for all of the analyzed Nb components of the billets. For the Nb elements of ITS01-08 strand it was shown that some difference between the grain sizes for Nb core and Nb tube occurred due to that elements had the different initial hot extrusion deformation degree. In the cold rolled Nb rods to be used in ITS02-08 strand non-uniform grain structure has been observed. However, in the cold drawed Nb rods for ITS15-08 strand very fine uniform grain structure has been formed.




(a) (b) (c)

**Figure 5.** Nb elements after recrystallization heat treatment: a – for ITS01-08 strand; b – for ITS02-08 strand; c – for ITS15-08 strand.

Second recrystallization heat treatment was carrying out on multifilament subelements before covering by tin layers and placing them in a final strand composite billet. The filament recrystallization structures are presented at Figure 6. The applied heat treatment was the same for all of multifilament subelements.



(a) (b) (c)

**Figure 6.** Filament restacks in subelements after recrystallization heat treatment: a – for ITS01-08 strand; b – for ITS02-08 strand; c – for ITS15-08 strand.

In the filament constructed from the cold drawing Nb rods for ITS15-08 strand the very fine uniform grain structure has been obtained as it is shown at Figure 7. Some large grains in this area have inner boundaries with low visible contrast, which possibly related to the subgrain structure.

After the second recrystallization heat treatment the multifilamentary subelements were covered by tin layers, assembled in final strand billets and deformed by cold drawing to final diameter of 0.82 mm.

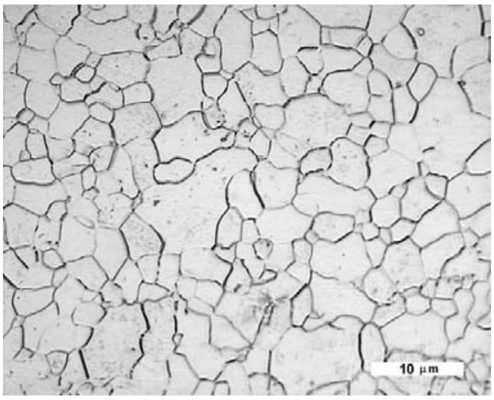



**Figure 7.** Nb grain structure in subelement filament of ITS15-08 strand.

### *3.2. Reaction heat treatment*

In order to study the kinetics of the $Nb_3Sn$ layer formation in the filaments of developed superconductors several different heat treatment regimes were applied for the strands at final diameter. The $Nb_3Sn$ layer thickness was measured from SEM micrographs after different duration and temperatures in a range of 650-800 $^oC$ that corresponded to the final stage of heat treatments applied. The ITS01-08 and ITS01-08 strands showed the similar kinetics of layer growth, but the strand ITS15-08 had the highest layer growth rate in a factor of 1.5 larger than witnessed in ITS01-08 and ITS01-08 strands.

The typical cross-section of the heat-treated superconductor strand (0.82 mm in diameter) is presented in Figure 8. Multi-stage reaction heat treatment for this strand was 210$^o$C (duration of 50 hours) + 340$^o$C (duration of 25 hours) + 450$^o$C (duration of 25 hours) + 575$^o$C (duration of 100 hours) + 650$^o$C (duration of 200 hours) with heating rate of 5$^o$C/h. The spectral SEM/EDS X-ray microprobe analysis shows that the residual tin distribution in matrix after reaction heat treatment for all investigated strands are uniform at the level of 4.5 wt.%Sn with 0.8 wt.% Mn.

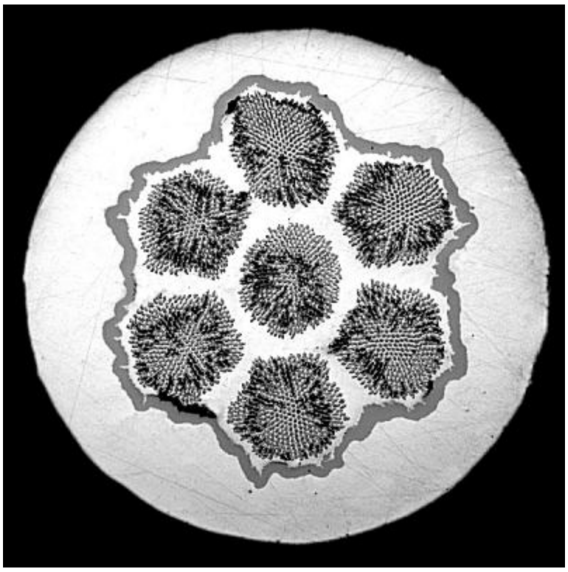
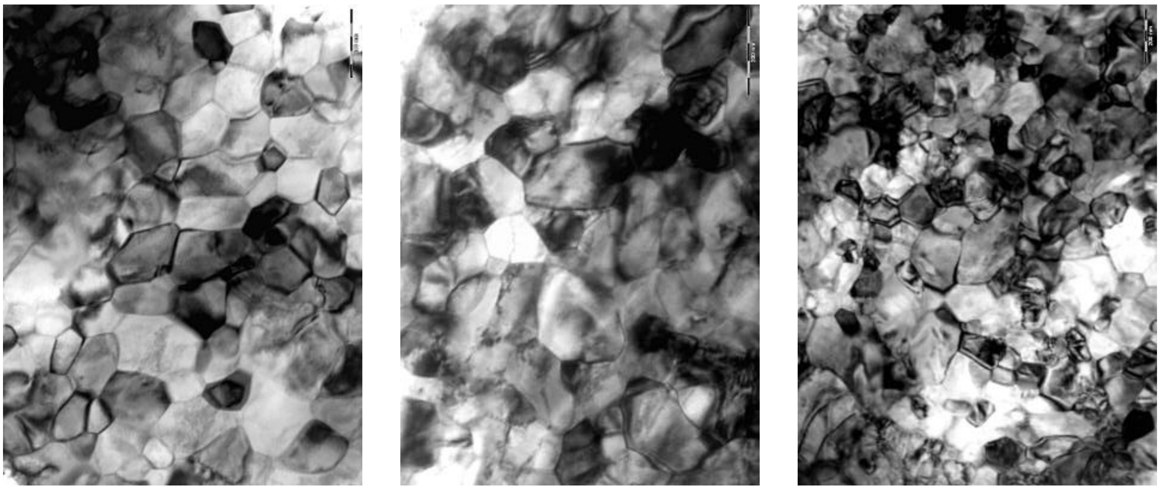


**Figure 8.** Typical cross-section of IT superconducting strand (0.82 mm in diameter) formed from round multifilamentary subelements after reaction heat treatment.

The grain structure of $Nb_3Sn$ phase in developed IT strands after multi-stage reaction heat treatment was studied by transmission electron microscopy (TEM). TEM images are presented at Figure 9. It should be stressed that for high quality $Nb_3Sn$ layers formation, the grain size and homogeneous of $Nb_3Sn$ phase are very important, because they determine in a final sense the pinning centers density and thus the critical current density as a final result [8].

At the Figure 9a the $Nb_3Sn$ grain structure is presented which characterized by the very large uniform grains. At the same time the grain structure with similarly large but essentially non-uniform grains, which was observed in the other sample of the strand, is presented at Figure 9b. The microstructure of the $Nb_3Sn$ layers for the third strand was characterized by the excellent fine and uniform grain structure that is illustrated by the Figure 9c. Judging from the above described TEM analysis results the best properties could be assigned to the ITS15-08 strand comparing to other two developed strands. It should be taken into account that the TEM analysis is local but in general the grain structure of ITS15-08 strand consisted of the regions with relatively uniform small equiaxed grains in a range of 90-150 nm.

The fine grain size (90-150 nm) observed for the strand ITS15-08 with composite filaments consisting of multiple niobium rods and rods made from NbTi alloy produced by cold drawing process was found to be smaller than for the strand sample with filaments consisting of niobium and NbTi rods formed by cold rolling (150-250 nm). This reduced grain size indicates a more efficient pinning, which explains the critical current density $J_c$ enhancement observed for the ITS15-08 strand.



(a) (b) (c)

**Figure 9.** Grain structure of $Nb_3Sn$ in IT strands after reaction heat treatment: a – in ITS01-08 strand; b – in ITS02-08 strand; c – in ITS15-08 strand.

*3.3. Measurement of strands properties*

The current-carrying capacity of strands was measured by four-contact technique at the temperature of 4.2 K in magnetic field of 12 T at the sensitivity of 0.1 and 1.0 μV/cm. The distance between the potential taps was 500 mm. The hysteresis losses of strands were measured by using the vibrating sample magnetometer technique at 4.2 K in the variable magnetic field of ± 3 T. Strands measurement results of critical current density at 12 T, 4.2 K and hysteresis losses are presented in Table 2. Results of critical current density measurements for IT strand as a function of magnetic field are presented at Figure 10.

**Table 2.** Properties of IT strands after reaction heat treatment.

| Properties | Strand identification | | |
|---|---|---|---|
| | ITS01-08 | ITS02-08 | ITS15-08 |
| Non-Cu $J_c$ (at 12 T, 4.2 K, 0.1 μV/cm), A/mm$^2$ | 740 | 815 | 860 |
| “n”-value | 23 | 27 | 29 |
| Hysteresis losses (on ± 3 T cycle at 4.2 K), kJ/m$^3$ | 300 | 350 | 370 |

From the table above, we can see, that round shaped subelements from our new approach allowed to stay at low level of hysteresis losses, because of filament bridging effect been reduced, simultaneously saved high level of critical current capacity for IT strands.

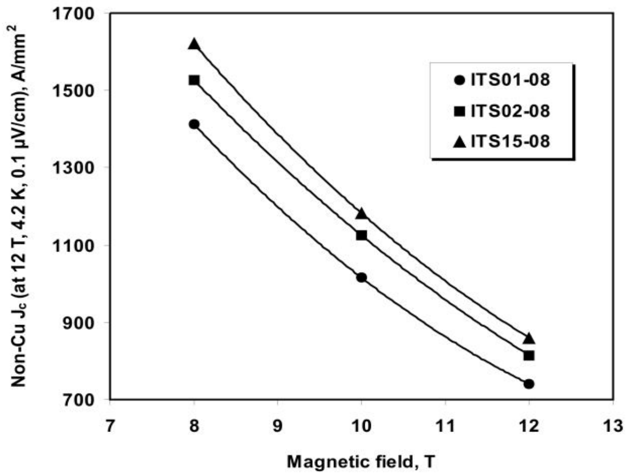



**Figure 10.** Non-Cu critical current density as a function of magnetic field for IT strands.

As expected, the best $J_c$ has ITS15-08 strand with fine grain size $Nb_3Sn$ phase. It is explained by inheritance of structure from initial Nb rods, formed by cold drawing deformation process.

The experimental results which are presented in this paper on the method of fabrication of the composite filament for IT $Nb_3Sn$ superconductors can be effectively used for industrial production of bronze process $Nb_3Sn$ strands for ITER also. This method does not require manufacture of special alloys for strand filaments, and suppose to use only industrially produced Nb and NbTi alloy for the appropriate superconductors type.

Furthermore, any additional equipments for components deformation process and composite billets preparation are not required. The tin content in a bronze matrix of bronze process superconductors is limited by the phase diagram. The enlargement of tin concentration in initial bronze ingots used for manufacture of superconductors by bronze process inevitably is accompanied by the increase of wire breaks during the deformation processing which in turn leads to a reduction of production yield. Thus the economic expedience of industrial production process is reduced. It was shown that the use of proposed composite filaments enabled to effectively regulate the grain size of niobium in multifilamentary subelements and as a consequence to increase more than on 15 % the critical current capacity for both IT $Nb_3Sn$ strands and bronze process $Nb_3Sn$ strand.

## 4. Conclusions

In this experimental work on the development of the ITER type IT $Nb_3Sn$ strands it has been proposed to use composite Nb filaments artificially doped by Ti which are formed from non-doped niobium rods in combination with the rods made of conventional Nb-47wt.%Ti alloy which is widely used in industrial production of Nb-Ti strands. It was shown that the formation of predetermined fine grain structure in Nb filaments could be effectively realized through the proposed design of the initial composite filament billet that is assembled from the set of Nb elements placed in a copper alloy matrix which are constructed out of separate Nb components subjected to the significant initial deformation for decreasing of Nb grain size.

Three types of $Nb_3Sn$ multifilamentary strands with distributed source of tin have been fabricated by internal-tin method with filaments formed by a new approach. After multi-stage reaction heat treatment the fine grain size (90-150 nm) of $Nb_3Sn$ phase was attained.

New approach to assemble of multifilamentary subelements in IT strands final composite billet was successfully tasted. In this approach the round shaped Cu-Nb subelements covered by tin layers was used instead of hexagonal shaped multifilamentary subelements as it was applied in the previous design. The positive influence of the use of round shaped multifilamentary subelements on the uniformity of filaments array in the final strands has been shown.

The superconducting properties of the IT $Nb_3Sn$ strand with fine grain size in filaments of round multifilamentary subelements was measured – the designed non-copper critical current density 860 A/mm$^2$ (4.2 K; 12 T) and hysteresis losses (±3 T) 370 mJ/cm$^3$ were attained.

It was shown that the use of proposed composite filaments enabled to effectively regulate the grain size of niobium in multifilamentary subelements and increased more than by 15 % the critical current capacity as for IT $Nb_3Sn$ strands and applicable for industrial bronze process $Nb_3Sn$ strand too.

## References

[1] Bruzzone P, Stepanov B, Wesche R, Salpietro E, Vostner A, Okuno K, Isono T, Takahashi Y, Kim H, Kim K, Shikov A, Sytnikov V 2008 Results of a new generation of ITER TF conductor samples in SULTAN *IEEE Trans. on Appl. Supercond.* **18** 459

[2] Twin A, Brown J, Domptail F, Bateman R, Harrison R, Lakrimi M, Melhem Z, Noonan P, Field M, Hong S, Marken K, Miao H, Parrell J, and Zhang Y 2007 Present and future applications for advanced superconducting materials in high field magnets *IEEE Trans. Appl. Supercond.* **17** 2295

[3] Vostner A, Bauer P, Wesche R, Vetrella U, Stepanov B, della Corte A, Portone A, Salpietro E, and Bruzzone P 2008 Qualification of the EFDA dipole high field conductor *IEEE Trans. Appl. Supercond.* **18** 544

[4] Field M, Parrell J, Zhang Y, Meinesz M, and Hong S 2008 Internal tin $Nb_3Sn$ conductors for particle accelerator and fusion applications *Adv. Cryo. Eng.* **54** 237

[5] Pantsyrnyi V, Shikov A, Vorobieva A, Silaev A, Soudiev S, Kozlenkova N, Beliakov N 2001 Superconducting properties of internal-tin $Nb_3Sn$ wire with enhanced current capacity *IEEE Trans. on Appl. Supercond.* **11** 3591

[6] Shikov A, Pantsyrny V, Vorobieva A, Sudyev S, Beliakov N, Sergeev V, Kozlenkova N, Polikarpova M 2009 Researches of Structure Formation Peculiarities in the Internal-Tin $Nb_3Sn$ Strands *IEEE Trans. on Appl. Supercond.* **19** 2560

[7] Shikov A, Pantsyrny V, Vorobieva A, Sudyev S, Sergeev V, Beliakov N, Kozlenkova N, Sytnikov V, Taran A, Rychagov A, Bruzzone P 2008 Development of a New RF Produced Internal Tin ITER TF Conductor Sample for Testing in SULTAN Facility *IEEE Trans. on Appl. Supercond.* **18** 1076

[8] Lee P J and Larbalestier D C 2008 Microstructural factors important for the development of high critical current density $Nb_3Sn$ strand *Cryogenics* **48** 283

## Acknowledgements

The authors are indebted to M.M. Potapenko, A.N. Zakharov, I.A. Panaschuk and G.A. Zakharova from Bochvar Institute for support and technical assistance. The technical support of the Moscow Electrical Engineering Institute (VEI) and Dr. V.F. Voloshin is greatly acknowledged. The authors wish to thank D.S. Anischuk, K.V. Utkin and A.V. Vorobieva for fruitful collaborative work in Chepetsky Mechanical Plant. This work was funded by State Atomic Energy Corporation "Rosatom".