**DEPARTMENT OF ENERGY
SMALL BUSINESS INNOVATION RESEARCH
SMALL BUSINESS TECHNOLOGY TRANSFER
RESEARCH GRANTS**

**Grant No. DE- SC0006235
Hyper Tech Research, Inc.**

Phase I Final Report

Increase J_c by Improving the Array of Nb₃Sn strands for Fusion Application

PREPARED BY:

***Dr. Xuan Peng, Principle Investigator*
Hyper Tech Research, Inc.
539 Industrial Mile Rd.
Columbus, OH 43228**

Subcontractor

***Dr. Mike Sumption, Subcontractor*
Dr. Edward Collings, Subcontractor
The Ohio State University
2041 College Rd
Columbus, OH 43012**

PHONE: (614) 481-8050 x2441

FAX: (614) 481-4080

Email: xpeng@hypertechresearch.com

Home Page: <http://www.hypertechresearch.com>

Grant period covering June. 17, 2011 to March. 16, 2012

Date of report: June 17, 2012

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ABSTRACT

During Phase I, our efforts were focusing on improving the array of subelement in the tube type strands by hardening the Sn core and the subelement matrix to effectively increase the J_c of the strands. Below is a summary of the results.

- 1) We were unsuccessful in improving the array using a Cu-Sn matrix approach.
- 2) We slightly improved the array using Sn with 1.5at%Ti doped core, and a 217-subelement restacked strand was made and drawn down without any breakage.
- 3) We greatly improved the array using the Glidcop Al-15 to replace the pure Cu sheath in the subelement, and a 217-subelement restacked strand was made and drawn down. Both strands have very good drawability and the array showed good improvement.
- 4) We also improved the array using improved wire drawing techniques using Hyper Tech's new caterpillar wire drawing machines to enable straight wire drawing for the entire wire drawing process.
- 5) The 919-subelement restack strand shows its non-Cu J_c over 2100 A/mm² at 12 T/4.2 K and AC loss of 508 mJ/cm³.

Phase I Results

The technical objective of the Phase I proposal was to improve the subelement shape and array to improve the non-Cu J_c s for advanced fusion conductor. It is the intent that the strands will have high J_c for 14-20T range magnets. The target is non-copper J_c s greater than 2400 A/mm² at 12T-4K on the wire (three times the present ITER specification at 12T) and over 1500 A/mm² at 15T-4K. In this Phase I we fabricated a couple of 217-subelement restacked billets with improved array, and finally selected one subelement with improved array to scale up to 919-subelement restacked billet. This 919-subelement restack strand has non-copper J_c s greater than 2100 A/mm² at 12T-4K and AC loss of about 500 mJ/cm³. The details are described in the following tasks.

Task 1. Harden the core of the subelements by using harder Sn alloy in the middle instead of pure Sn, and make two 217-subelement restacks.

As we increase the number of filaments the J_c usually reduces because of the distortion of the filament area. Ideally each filament is hexed, in the outer edges, with a round solid Sn core. The reaction takes place in a round format as the round Sn diffuses in the hexed Nb7.5wt%Ta tube. The Sn reaction should stop just short of the flats of the hexed shape. This leaves around 20-25% of the filament Nb cross section unreacted. The more distortion in the subelements, to maintain RRR (by avoiding Sn leakage), the lower will be the potential J_c value. One reason for the increased distortion as the filaments get smaller is thought to be a result of the soft Sn core. Therefore we want to experiment with approaches to harden up the Sn core.

Subtask 1: One way we hardened the core is to make the present core (a Cu tube with Sn inside) into a metal matrix composite.

We made the core a multifilament wire of Cu tubes with pure solid 100% Sn. We started with the Cu-Sn bundle. Firstly we inserted the pure Sn rod (0.4375" OD) in 0.015" wall, 0.56" OD Cu tube, then we drew the assembly down to about 2.605 mm, using 10% AR, as shown in Figure 1 (a). The Cu-Sn piece was cut into 19 pieces of about 36" long, and the 19 pieces were then stacked into a 0.58" OD x 0.53" ID Cu tube as a restack. This restacked bundle was drawn down to about 5.456 mm as shown in Figure 1 (b), which was then inserted into a Nb and Cu tube to become a subelement. Figure 2(a) shows the concept. The assembled billet was drawn down to 1.12 mm and then hexed through the 0.0382" F-F hexagonal die, Figure 2(b) is the cross-section of the final hexagonal subelement ready for the 217-pattern restack in 3/4" Cu tube.

(a) Cu-Sn assembly

(b) the hardened Cu-Sn core

Figure 1. Hardened Cu-Sn core for subelement.

(a) Tube type subelement structure

(b) the hardened Cu-Sn core

Figure 2. Hardened Core subelement.

This hardened core subelements were restacked into a special ID Cu tube as shown in Figure 3 (a). The billet was denoted as T2589 and it was drawn down. Figure 3 (b) shows the cross-section of the restacked wire at 2 mm. This wire was drawn down to 0.7 mm, but it experienced a couple of breakages at about 1.0 mm. The breakage starts with the Cu breakage in the subelement core. We will not pursue this in our Phase II.

Figure 3. (a) Customized ID Cu tube for 217-subelement pattern restack; (b) Cross-section of T2589 at 2 mm.

Subtask 2: We proposed another way to harden the Sn core would be to use Sn3%Ta alloy in the core. However during the Phase I, Professor Tachikawa was unable to deliver Sn3%Ta as we had originally planned. We also tried to have the Sn3%Ta alloy made for us by Ames Lab, but they could not make a Sn3%Ta with small enough precipitates. However, Ames Lab has made for us an alloy of Sn doped with 1.5at%Ti. We have shown in the past that this level of Ti in Sn is effective in raising J_c at 15-18 Telsa when used with Nb7.5wt%Ta subelements. Thus we wanted to see the effect on the array for this tubular approach wire. We used this alloy, replacing pure Sn, and made a subelement (as shown in Figure 4a), and then drew it down to get ready for a 217 restack.

Figure 4 (a) **Left:** Filaments showing the Sn with 1.5at%Ti alloy center instead of pure Sn. (b) **Right:** Cross section at 0.7 mm restacked strand.

The subelements were restacked into a $\frac{3}{4}$ " OD Cu tube. This billet was denoted as T2631. The billet was drawn down to 0.7 mm without any breakage along the process. Figure 4b shows the cross section of the 0.7 mm wire. The strand was sent to OSU for characterization. The Ti additive is supposed to refine the grain size and thereby increase the non-Cu J_c . We feel that the array showed a slight improvement, enough that this approach should be further explored in the new proposed Phase II. We will draw the strand down to 0.5 mm, 0.325 mm, and 0.25 mm which produced filament sizes of 25 μm , 16 μm , and 12 μm , respectively. In Phase II, we will scale this approach up to 919-subelement billet which will give filament size of 18 μm at 0.82 mm, and even further to determine the improvement of the array and the potential improvement of J_c at the higher fields of 15-18 Tesla at 4.2K.

Task 2. Harden the matrix between subelements by using the Cu-alloy in the subelements instead of pure Cu

Usually we use pure OFC Cu for subelement sheath, which has excellent thermal and electrical conductivity, and the OFC Cu is softer and easier to be drawn down. All the subelements are bundled together in the restacked wire and drawn down. As the subelements get smaller, there is more distortion of the subelements, and it becomes more difficult to maintain RRR (avoid Sn leakage), thus there will be the potential for lower J_c values. The soft Cu between subelements and the amount of this soft Cu is another effect leading to the distortion. Hence, in this task, we selected Glidcop Al-15 as the subelement sheath material instead of OFH Cu. Glidcop Al-15 is mechanically stronger. Glidcop Al-15 is much stronger than OFH Cu, but their thermal and electrical conductivities are similar. Figure 5 shows the design structure of the subelement and its 217-subelement restacked strand. This restacked strand was denoted as T2745. In Glidcop, the addition of small amounts of aluminum oxide increases the strength of the alloy, while keeping its high thermal conductivity and electrical conductivity. The addition of aluminum oxide also increases resistance to radiation damage. The heat during the drawing process will not soften the Glidcop, which will help to keep the shape of the subelement array.

(a) T 2745 Subelement with Glidcop

(b) T2745: 217 restacked strand at big diameter

Figure 5. Filament showing the Glidcop sheath instead of OFHC Cu

Figure 6. 0.7 mm strands cross-sections. Left: Glidcop between subelements; Right: OFH Cu between subelements.

This strand was drawn down to 0.7 mm. Figure 6 left is the cross-section of the 0.7 mm wire of T2745. Figure 6 right shows the similar design 217-subelement strand with OFH Cu between subelements at 0.7 mm. Upon comparing both pictures, we see that T2745 has relatively better subelement array. In order to show its benefit, we are proposing to scale up the billet to 919-subelement in this new proposed Phase II. We heat-treated the 0.7 mm strand of T2745, and OSU tested its transport properties. Figure 7 shows its J_c curve. The J_c is quite low. We will look at the cross-sections and investigate the reason further in the Phase II.

Figure 7. non-copper J_c curve of T2745 with field.

Task 3. Use the best technique above or a combination, and make a 919-subelement restacked billet. This restack will give filament size of 18 microns at 0.82mm.

Through Task 1 and Task 2, we were trying to harden the subelement core and subelement matrix to improve the subelement array in the final restack. Both the 1.5at%Ti added strand and the Glidcop sheathed strand could be drawn down very easily and with no wire breakage. The Glidcop sheathed strand was drawn down to 0.7 mm, 0.5 mm, 0.325 mm, and 0.25 mm, which creates subelement sizes of 35 μm , 24 μm , 16 μm , and 12 μm , respectively. In the Phase I, due to time limitation, we did not have the chance to make a high count restack using this subelement. But in this proposed Phase II effort, we plan to make higher count restack using this subelement.

We continued working on 919-subelement restack of regular subelement. We made regular subelements and drew it down to the restack size, hexed, and then restacked them in a 1.5" OD Cu tube with 919 filaments. This billet was denoted as T1791S. We drew the wire down to 0.82mm which gives a filament size of 18 microns.

In order to avoid the subelement distortion in the outer ring, we used an extra outer row of hexed filaments except they were 100% Cu filaments instead of normal filaments (Cu clad Nb7.5wt%Ta tubes with Cu/Sn inside) shown in Figure 8. This design gave 744 Nb7.5%Ta-Cu filaments. The restacked strand were drawn down to 0.875 mm and then twisted with pitch length of 20 mm, and then overdrawn down to 0.82 mm. This strand was heat-treated at 625 $^{\circ}\text{C}$ for 84 hours. Figure 9 shows its cross-section before and after heat-treatment.

Figure 8. (744+175) subelement design.

Figure 9. Cross-sections of the 919-subelement restacked strand at 0.82 mm. (Left) before and (right) after heat treatment.

OSU tested its transport properties and AC loss. Figure 10 shows its non-Cu J_c curve with field. Its non-Cu J_c at 12 T reaches at about 2125 A/mm². Figure 11 shows its magnetization curve. It shows a very smooth curve and no flux jump showed. Its AC loss is 508 mJ/cm³, which is far below the current fusion spec. This result demonstrated that the 919-subelement restack is good enough to meet the AC loss requirement of future fusion conductor. Hence, in this Phase II, we will focus on improving the non-copper J_c at high field and making long length strands of 919-subelement restack billets.

Figure 10. Non-Cu J_c at 0.82 mm as measured at OSU.

Figure 11. Magnetization curve of T1791S.

Task 4. Improve drawing technique to reduce distortion

Drawing wire straight (not putting around a circular bull block while drawing) is another way to reduce subelement distortion in the final small size wire. Due to the length limit of the straight drawing bench, we normally employ the circular bullblock approximately 1 meter in diameter to draw the wire starting at about 4.5 mm. The bending of big diameter wire evidently creates a non-uniformity issue. Figure 12 (left) shows a cross-section at 4 mm which has been drawn through the circular bullblock at 4.5 mm. From its cross-section, one can see the bending effect very clearly, one side is in tension and the opposite side is in compression, which has damaged the subelement array and also the bonding between the subelements. Hyper Tech Research has built and installed in 2011 caterpillar drives to draw wire straight without length limitation. Figure 13 shows one of these machines. We changed our drawing schedule and decided to draw the strand straight down to 2.3 mm in the caterpillar drive, and then transfer to bullblock, 0.5 meter diameter. Figure 12 (right) shows the cross-section of a similar strand with less Cu on the outside drawn down using the caterpillar drive by the same drawing schedule. The wire is also 4 mm. The subelements are evenly distributed. Both wire have been drawn down to final size, during the drawing process the left wire had breakage problem while the right one has no issues with drawing. Hence, we started using the Caterpillar drive for wire drawing straight to the final size for our regular high count restacked strands.

Figure 12. Wire cross-section showing the distortion during bullblock drawing at big size. Left: strand cross-section drawn with circular bullblock drawing at big size; Right: strand cross-section drawn with caterpillar drive drawn to small size.

Figure 13. One of four caterpillar drives built and installed at Hyper Tech Research in 2011.

Task 5. Characterization of restacked strands including SEM observations, transport properties, RRR testing and AC loss testing.

All the test results have been shown in Tasks 1-4, below we describe the types of tests and how they are conducted.

Barrel Winding and Reaction Heat Treatment: Segments of strand were wound onto ITER barrels in the standard way, and given various heat treatments. The samples were heat treated under argon and then soldered down to the barrels in the usual way (in which Cu end-rings are attached throughout the process). Reaction heat treatments were performed on test segments of strand at 0.82 mm.

Superconducting Property Characterization: The important measurements that were performed are the transport J_c measurements of the restacked strands wound on ITER barrels. The measurement set-up has a fast quench protection circuit, and the measurement probe is 1800 A-capable. $M-H$ loops were taken with the 9 T VSM and PPMS to investigate both the level of flux jumping and the overall AC loss (and, of course, d_{eff}). These properties are of course correlated with the diffusion of Sn and the effectiveness of not losing Sn, and these correlations were made with SEM and optical analyses.

Electro-optical Characterization: For this work there are two main tasks: (1) determination of the level of reaction completeness, and (2) checking the chemistry of the subelements and strands.

RRR Testing: RRR results are used to judge the reaction of the strands, and if the matrix is contaminated by Sn.

AC Loss Testing: Strands are wound on small screws in the standard way and given various heat treatments. The AC loss samples are tested in the PPMS machine at OSU.

Summary

During Phase I, our efforts were focusing on improving the array of subelement in the tube type strands by hardening the Sn core and the subelement matrix to effectively increase the J_c of the strands. Below is a summary of the results.

- 6) We were unsuccessful in improving the array using a Cu-Sn matrix approach.
- 7) We slightly improved the array using Sn with 1.5at%Ti doped core, and a 217-subelement restacked strand was made and drawn down without any breakage.
- 8) We greatly improved the array using the Glidcop Al-15 to replace the pure Cu sheath in the subelement, and a 217-subelement restacked strand was made and drawn down. Both strands have very good drawability and the array showed good improvement.
- 9) We also improved the array using improved wire drawing techniques using Hyper Tech's new caterpillar wire drawing machines to enable straight wire drawing for the entire wire drawing process.
- 10) The 919-subelement restack strand shows its non-Cu J_c over 2100 A/mm² at 12 T/4.2 K and AC loss of 508 mJ/cm³.

As we progress to the proposed Phase II program, we will continue working on 1.5at%Ti doped Sn billet and Glidcop Al-15 sheathed billets, and scale up the billet from 3/4 inch to 2 inch diameter billet restacks. We will focus on the 919 count restack and develop a long length strand for application. The details of our Phase II plans are given below.

PHASE II PROJECT

Technical Objectives

This Phase II is aimed at continuously improving the subelement array to improve the non-Cu J_c s and reduce its AC loss for use in advanced fusion conductor. The target is non-copper J_c s greater than 2500 A/mm² at 12T-4K on the wire (three times the present ITER specification at 12T) and 1500 A/mm² at 15T-4K with the small filaments and low AC losses. Through the Phase I effort, we demonstrated that 919 count restack strand at 0.82 mm meets the future fusion conductor requirement, which has AC loss of 508 mJ/cm³ while keeping non-Cu J_c over 2100 A/mm² at 12 T/4.2 K. Hence, in this Phase II, we expect to continue to improve its non-Cu J_c in 919-subelement restacked strand through improving the array, doping Ti and also the drawing technique and improve the piece length of the 919-subelement restacked strands at 0.82 mm. The work details are described in the following tasks.

- Task 1. Continue working on the 1.5at%Ti doped Sn core billet and find its limit on wire drawability, array improvement and J_c improvement at 15T.*
- Task 2. Billet 1: Make a 919-subelement restack using the subelement with 1.5at%Ti doped Sn core.*
- Task 3. Billet 2: Use Glidcop Al-15 as the subelement sheath and scale up to 919-subelement restack.*
- Task 4. Billet 3: Replace the pure Cu with Cu alloy (Glidcop Al-60) in the core of subelement to introduce fine precipitates in the Nb-Sn interface to refine the grain size, and improve its non-Cu J_c . A 217-subelement restack will be made to test its properties.*
- Task 5. Billet 4: A 919-subelement restack will be made using the subelement design from Task 4.*
- Task 6. Billet 5: Use our best filament design (combination of improvements) to construct another 919-subelement restacked billet.*
- Task 7. Continue to improving the assembly and drawing technique to develop the 5-6 Km length of 919-subelement restack at 0.82 mm.*
- Task 8. Characterize the restacked strands at OSU.*
- Task 9. Assess Strain Tolerance at NIST.*
- Task 10. Produce enough strands for evaluation and testing.*