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

Grant No. DE- SC0006254
Hyper Tech Research, Inc.

Phase I Final Report

Increasing the J_c of Tube-Type Nb_3Sn Strands

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: March 17, 2012

ABSTRACT

In this Phase I, we successfully made strands with better Cu/Sn ratio to reduce the coarse Nb_3Sn grain region, thereby providing the potential of increasing the non-Cu J_c in the Phase II and scaling up to 2” billets with 331 subelements. In order to improve the strand’s high field properties, we successfully doped low amount of Ti in the subelements and made a 217-subelement wire which has been drawn down to 0.7 mm without any breakage. This strand gave subelement size of 35 µm. We will scale up the Ti-doped billet to 271-subelement in 1.5” billet in this proposed Phase II. The hexagonal shaped subelements with round Nb-Sn have been developed for a 61-subelement restack. Thus the results indicated that for 217-subelement restack in a 2” billet we have the potential to draw down this type of construction without problems while maintaining a good array to react more Nb to get higher non-Cu J_c in the Phase II.

Phase I Results

In the Phase I, we have demonstrated we can improve the strand and increase the non-Cu J_c of the tube type strands with subelement size of 30-35 µm through improving the Nb/Sn, Cu/Sn ratios, adding small amount of Ti in the Sn, and improving the subelement shape to react more Nb. All the details in each task are described as follows.

Task 1. Further optimization of Nb/Sn ratios, Cu/Sn ratios, and optimized heat treatment especially at more aggressive heat treatment schedules to target 15T plus fields, and obtain optimized J_c while maintaining RRR (30-100) to obtain high J_s at low fields for stability. This has been done with 217 subelement restack with d_{eff} in the 30-35 µm range.

To maintain a good RRR and avoid Sn leakage into the Cu matrix we have varied the Nb/Sn ratios from 3.0 to 3.2 in the 217-subelement restacked strands. The strand with Nb/Sn ratio of 3.2 has high RRR with non-Cu J_c of 2100 A/mm^2 while keeping high stability down to zero Tesla (high I_s and J_s at low fields). In order to increase the J_c to 2500 A/mm^2 or higher while keeping this design with high RRR, we needed to pursue other ways. In this task, we focused on changing the Cu (inner tube)/Sn ratio in the middle to reduce the formation of Nb_6Sn_5 thereby reducing the coarse grain region converted from Nb_6Sn_5.

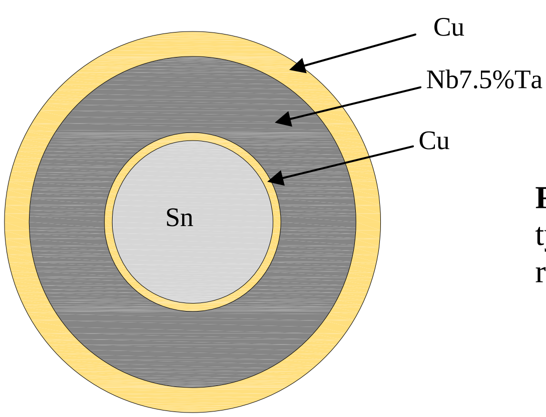


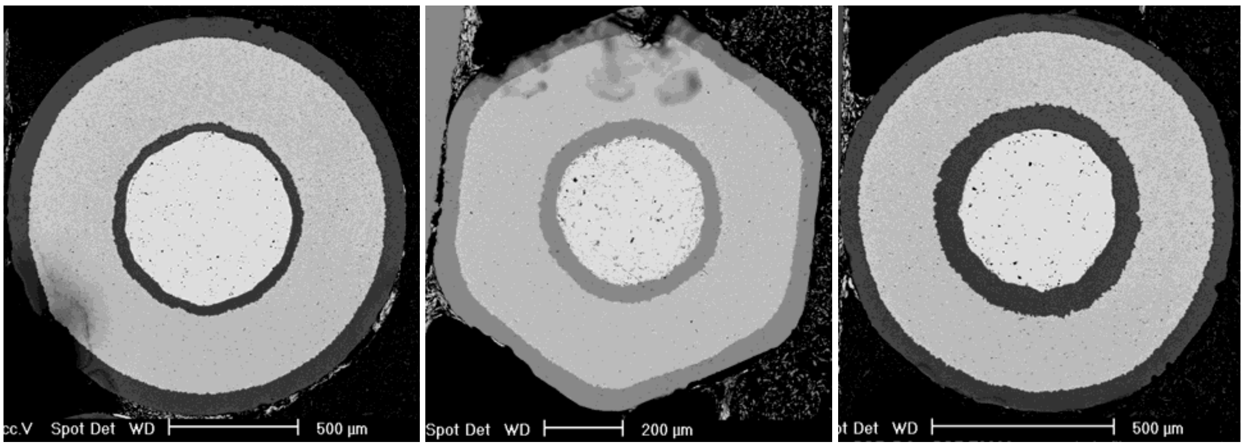
Figure 1. The subelement structure for tube type strand. Varying the Cu (inner tube)/Sn ratio in the center is investigated in this task.

As shown in Figure 1, we varied the Cu (inner tube)/Sn ratio in the subelement center through using different wall thickness Cu tube around Sn. We made three subelements using different wall thickness Cu tube and then stacked the subelements to make three 217-subelement restacked strands separately. Table I shows the design parameters for the three conductors.

Table I. Design parameters for the three conductors with various Cu (inner tube)/Sn ratios.

Conductor Name	Nb/Sn ratio	Cu/Sn ratio	Cu tube in the center
T2484	3.15	0.253	3/8” OD x 0.020” thickness
T2637	3.13	0.369	0.53” ID x 0.62” OD
T2639	3.08	0.512	3/8” OD x 0.035” thickness

In T2484, the 3/8” OD x 0.020” thickness tube was used, in T2637, the 0.62” OD x 0.53” ID tube was used, and in T2639, the 3/8” OD x 0.035” thickness tube was used. Their Nb/Sn ratios have little difference.



(a) T2484 subelement (b) T2637 subelement (c) T2639 subelement

Figure 2. The subelement cross-sections showing various Cu (inner tube) /Sn ratios.

All the three subelements were drawn down to 1.12 mm and then went through the 0.0382” F-F hexagonal die to shape the subelement. Figure 2 shows their cross-section at 1.12 mm or after hexing through the 0.0382” die. The hexed subelements were cut into pieces with 5 ft lengths. All the pieces were restacked into a customized ID, ¾” OD Cu tube as shown in Figure 3. The ridged customized ID tube perfectly held the array of subelements.

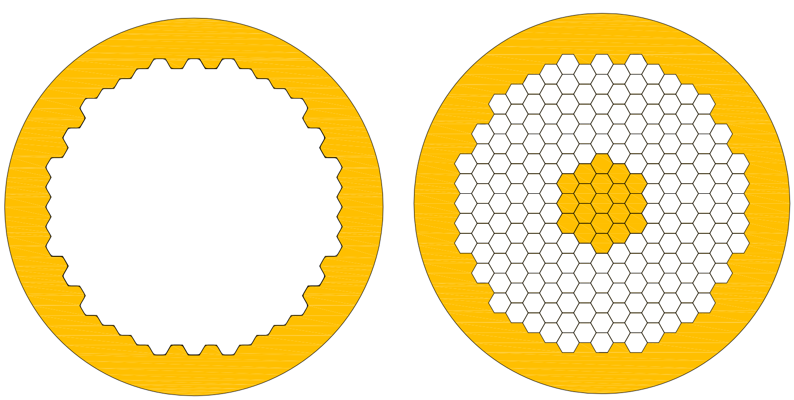
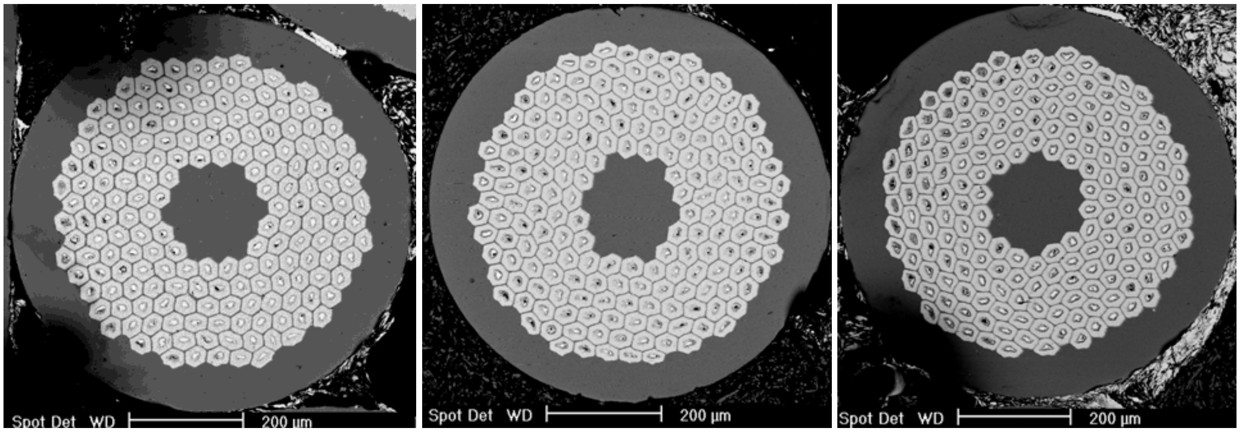


Figure 3. Schematic drawing of 217-subelement restack, customized tube and restack.

All the three 217 restacked strands were drawn down to 0.7 mm, Figure 4 shows their cross-sections. All the strands were heat-treated under 625 °C for different times. Table II shows the heat-treatment schedule.



(a) T2484 at 0.7 mm (b) T2637 at 0.7 mm (c) T2639 at 0.7 mm

Figure 4. Cross-sections of the different strands at 0.7 mm with different Cu/Sn ratios.

Table II. Heat-treatment schedules of the strands.

t (hour) / T (°C)	T2484	T2637	T2639
625	100	100	100
	150	150	150
	200	200	200
	250	250	250
	300	300	300

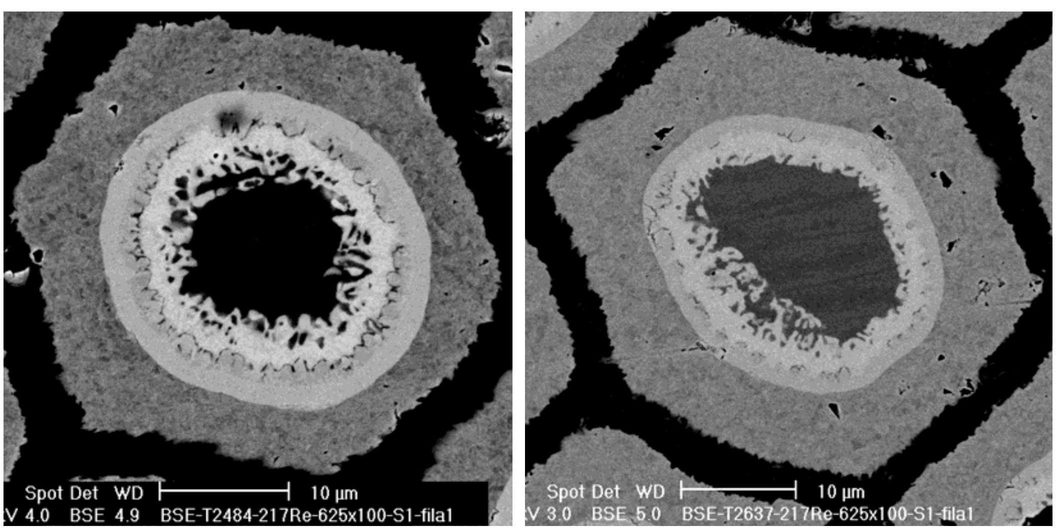


Figure 5. Cross-sections of T2484 and T2637 after heat treating at 625 ^{0}C for 100 hr.

OSU characterized the heat-treated samples under SEM observation. The samples of T2639, with the highest Cu/Sn ratio, have not been characterized yet. Figure 5 shows the results of reaction after 625 ^{0}C for 100 hours of T2484 and T2637. In both strands A15 started to form. From inside out, there is an Nb_6Sn_5 layer, then a coarse grain Nb_3Sn region, and then a fine grain Nb_3Sn region. Comparing both strands, the Nb_6Sn_5 layer is thinner in T2637 which may be due to more Cu involved there. Figure 6 shows the reacted samples after 250 hours annealing. It can be seen that the T2637 has more uniformly reacted subelements, and there is less coarse grain Nb_3Sn region. But overall there is less Nb_3Sn formed. Comparing these two samples, putting more Cu in the Cu-Sn center could reduce the Nb_6Sn_5 region thereby reducing the coarse Nb_3Sn region. OSU is testing these strands' transport properties. In the proposed Phase II, we will also use T2367 design but will add more Sn to react more Nb to improve its non-Cu J_c.

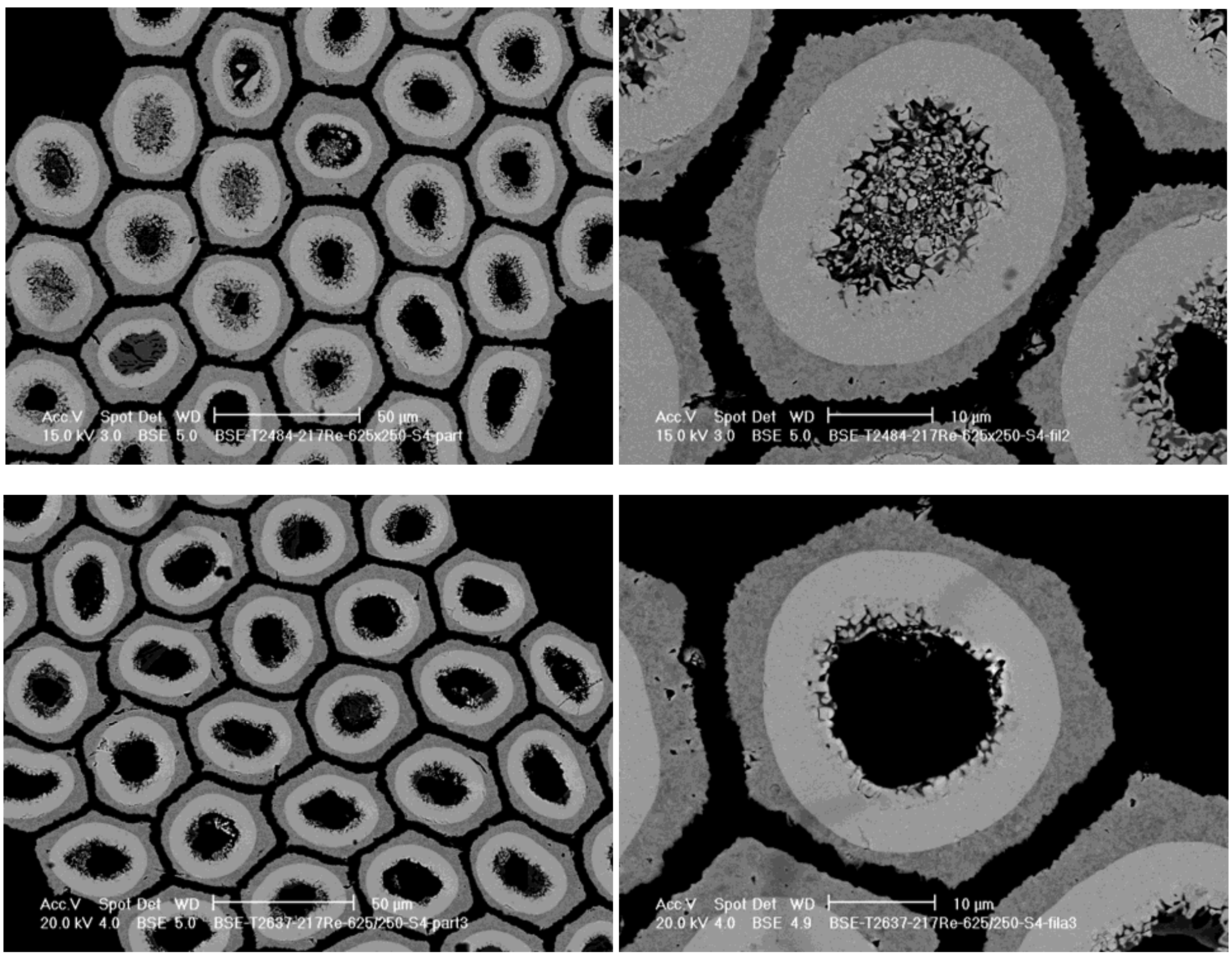


Figure 6. Cross-sections of T2484 and T2637 after heat treating at 625 ^{0}C for 250 hr.

Task 2. Make subelements with improved tri-layer barrier around the subelement to stop Sn leakage and maximize the reaction of available Nb. This will increase the non-Cu J_c by reacting all the Nb while the barrier is blocking the Sn leaking.

In this task, we used a barrier to stop Sn leakage so that all the Nb inside the barrier could be reacted, which could increase the non-Cu J_c. Furthermore, this special barrier will also strengthen the strand itself and may increase its strain tolerance. Through previous SBIR Phase I, we have already demonstrated that a specially designed sandwich barrier (tri-layer) could be used and the special barrier was drawable in a 61 restack, indicating that the barrier can be drawn down in diameter to achieve 30 μm and less subelement size. We adapted this technique and made a 217-subelement restacked strand. This strand was denoted as T2752. Figure 7 shows the subelement structure of T2752.

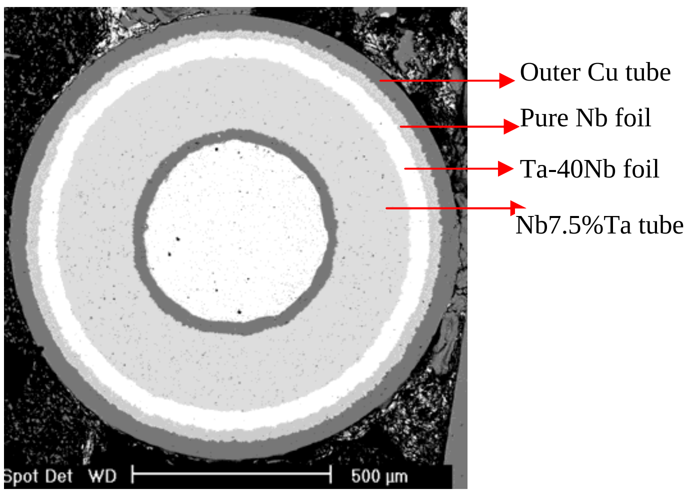


Figure 7. The subelement structure with tri-layer barrier of T2752 (Nb40Ta60 foil as a barrier backed up with pure Nb foil creating a tri-layer structure).

The starting subelement was constructed with an outer barrier consisting of a 0.015" thick Nb40Ta foil backed up with 0.010" thick pure Nb foil. The standard Cu/Sn ratio of 0.253 was used in T2752. Nb/Sn ratio of 2 was used to fully react the subelement with a high Sn content. The subelement was drawn to restack size and hexed in the final passes. The subelement was then restacked into a customized Cu tube. This billet has (180+19) subelements. Figure 8 shows its cross-section at 2 mm. The billet was drawn down, but has breaks at about 1 mm which has subelement size of about 50 μm. Due to the drawing issues, we will not pursue this method in this Phase II,

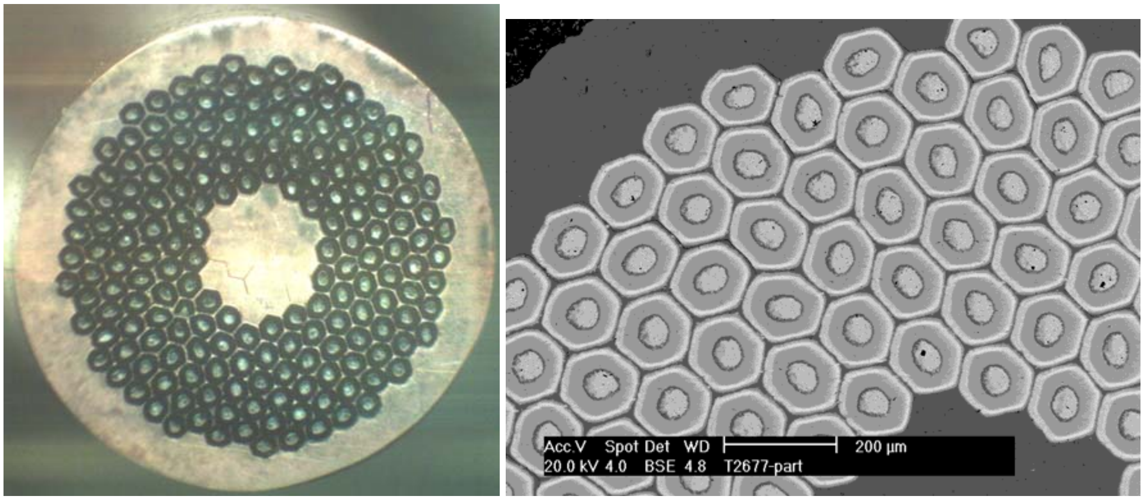


Figure 8. Cross-section of T2752 at 2 mm.

Task 3. Make special subelements with hexagonal Cu on outside and round Nb inside to maximize the reaction of the available Nb. This can increase the non-Cu J_c to 3000 A/mm^2 at 12T-4K.

Recent comparison study on tube type and internal-tin strands indicated that both strands have similar layer J_c while the non-Cu J_c of the tube type strand is lower than that of internal-tin strand. The reason is that not all of the hexagonal Nb (corners) can be reacted. In order to convert as much as possible of the Nb to an A-15 compound before the matrix becomes contaminated with Sn, the ideal condition will be that the outside of the Nb alloy tube is round and concentric with the Sn core - a condition that occurs and can be maintained in a single subelement. However, when a restack is made, the outside of the Nb alloy tube (subelement) is naturally hexagonal. Thus, A15 growth reaches the flat surfaces of the hexagon first and immediately the Sn begins to leak into the Cu matrix, destroying the RRR of the matrix, leading to instabilities.

In this task, we started with ¾” Cu OD tube, and tried to get Cu “tube” with a round ID and a hexagonal OD for subelement. For 217-subelement in ¾” tube, the subelement size is about 1 mm which is too small to get the tube made. Hence, we reduced the restack count to 61-subelement, which requires the subelement size of about 2 mm. Figure 9 is the plot for the 61-subelement restack where the dark circles are the round subelements with Nb outer wall.

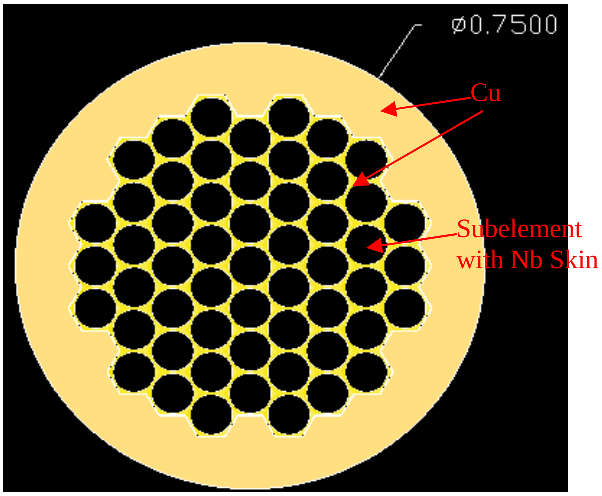


Figure 9. Schematic plot of 61-subelement with perfect array.

We purchased the Cu "tube" with a round ID and a hexagonal OD from AT Wall for this experiment. The copper tube dimension was 0.071" F-F OD x 0.046 ID, which is the best we could get in this size of tube. The thickness is about 0.0125" which indicates the S/D is up to 0.5. It is very high. In order to match our standard subelement with 0.58" OD x 0.53" ID, we had to etch the tube. It is hard, but we got it done. Then we made a standard subelement, and drew it down to 1.2 mm, then etched the outer Cu away. The subelement with Nb on the surface was assembled into the Cu tube. Figure 10 shows the assembled subelement.

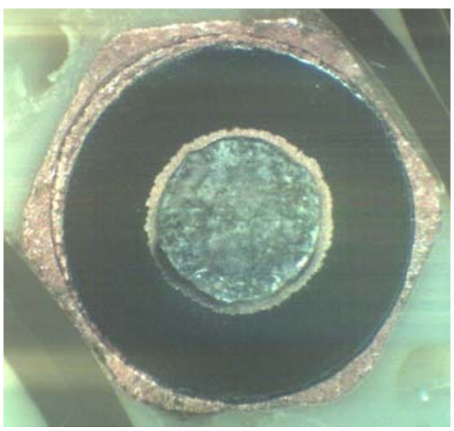


Figure 10. Assembled subelements showing hexed Cu (outside) and round Nb (middle).

We assembled 54 hexagonal Cu round Nb subelement and 7 Cu filaments and restacked them in the Cu tube, and drew down the restacked billet. This restacked wire was denoted as T2677. Figure 11 shows the cross-sections of T2677 at 1 mm and 0.7 mm. It is obvious that there is too little Cu between the subelements in this restacked strand. We need to leave more Cu in the next subelement.

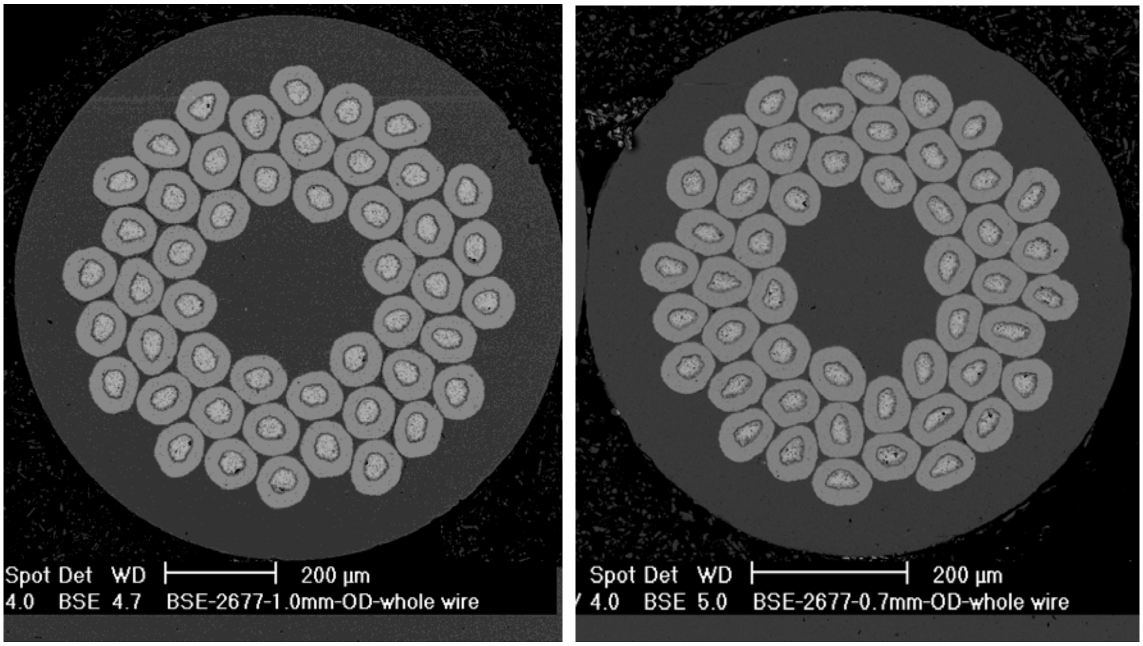


Figure 11. Cross-sections of T2677 at 1 mm and 0.7 mm.

We heat-treated the 0.7 mm samples of T2677 at different schedule. Figure 12 shows their reactions. It could be seen clearly that more Nb could be reacted by keeping the subelement round. In addition, we could add more Sn to react more Nb to increase the non-Cu J_c.

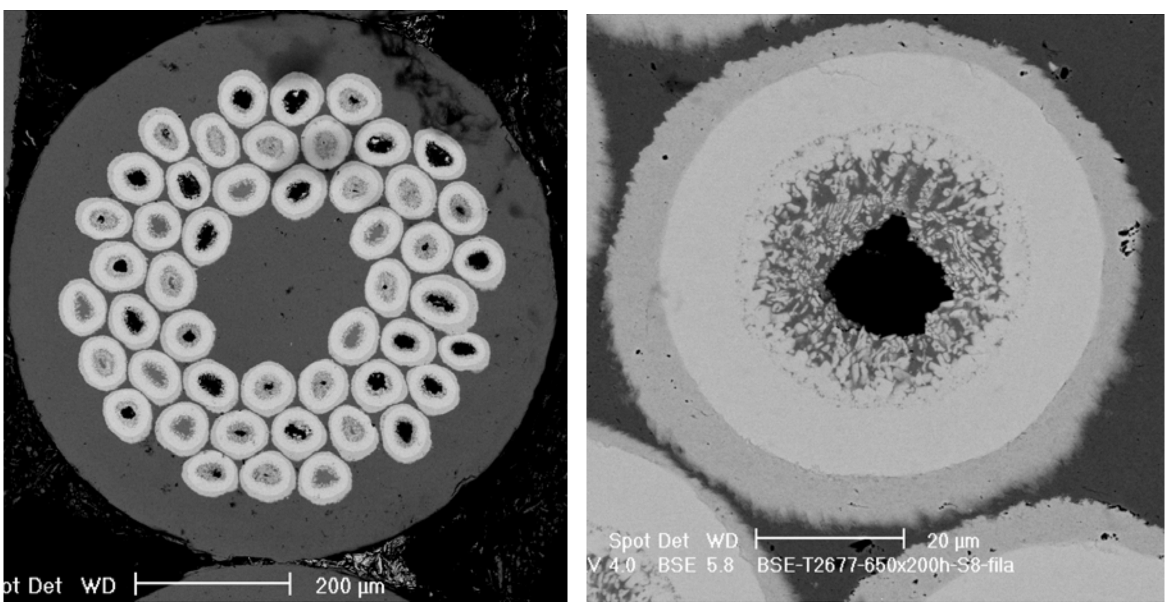


Figure 12. Cross-sections of reacted samples of T2677.

In order to increase the Cu spacing between the subelements, we did another experiment on using the hex OD round ID Cu tube. We etched the Cu tube to get the S/D of 0.2. We made a standard subelement as in T2677, and drew it down to 1.2 mm, then etched the outer Cu away.

The subelement with Nb on the surface was assembled with the Cu tube. This subelement was assembled in the 61-subelement customized ID Cu tube. This restack wire was denoted as T2716. The restack was drawn down. Figure 13 shows its cross-sections at 4 mm and 0.7 mm. In the 4 mm cross-section, the subelement shape is pretty decent, but it has more distortion when drawn down to 0.7 mm. We are not sure if it is due to the end effect or real distortion in the wire. We will continue to investigate this in Phase II. This strand was heat-treated and samples were sent to OSU for characterization including J_c testing. In the phase II, we will scale up the billet size to 2" OD, and subelement count to 217, which will increase the stacking subelement size to about 3 mm. In addition, we will adjust the Cu spacing between the subelement in T2677 and T2716 to reduce the subelement distortion during drawing process.

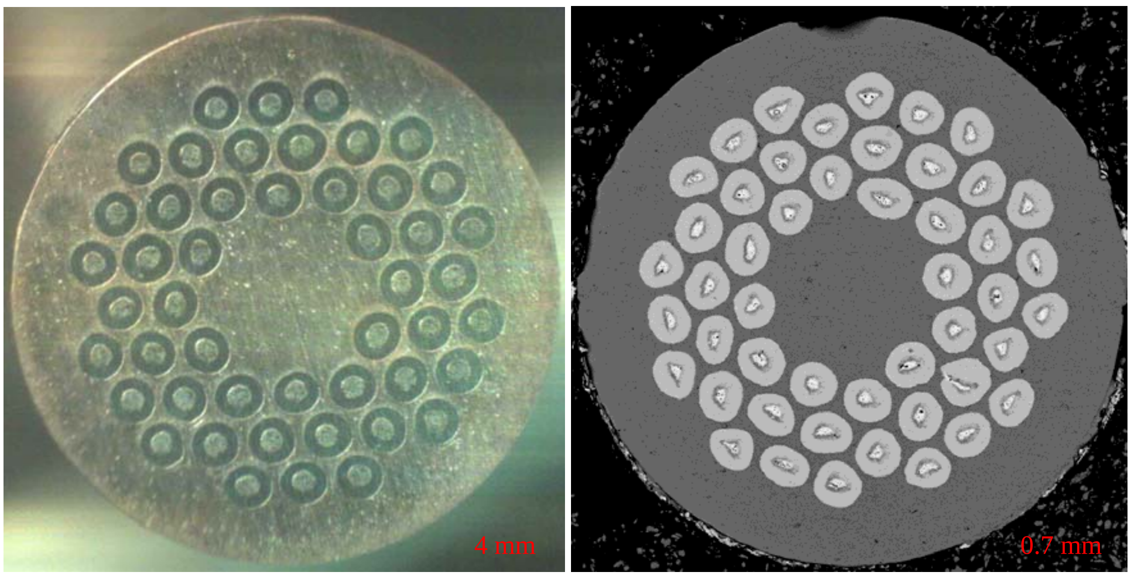


Figure 13. Cross-sections of T2716.

Task 4. We need to look at approaches to increase the non-Cu J_c of Nb_3Sn at high fields, such as a change of chemistry, an additive that affects pinning and/or B_{c2}. We doped with a small amount of Ti in the billet. The billets with Ti-doped could also be used to investigate the effect of Ti addition on strain properties of final strand.

In this task, we made a strand with Nb7.5%Ta doped with small amount of Ti. Ames' Lab made a Sn alloy doped with 1.5at%Ti for us. This Sn alloy instead of pure Sn was used in the middle as shown in Figure 14. This task was performed because from previous work with RIT type strands we have shown that doping Nb7.5wt%Ta with 1.5at%Ti had improved the J_c at 15T, so we believe the same result can be achieved in the Tube Type strands.

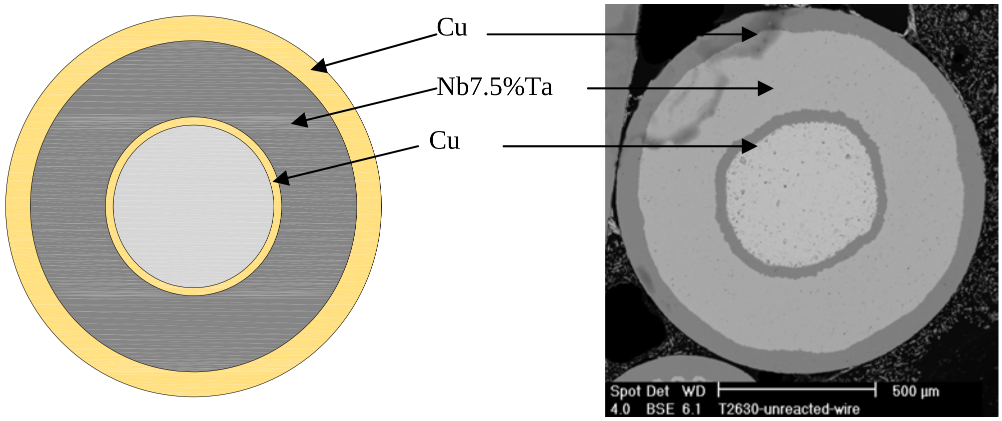
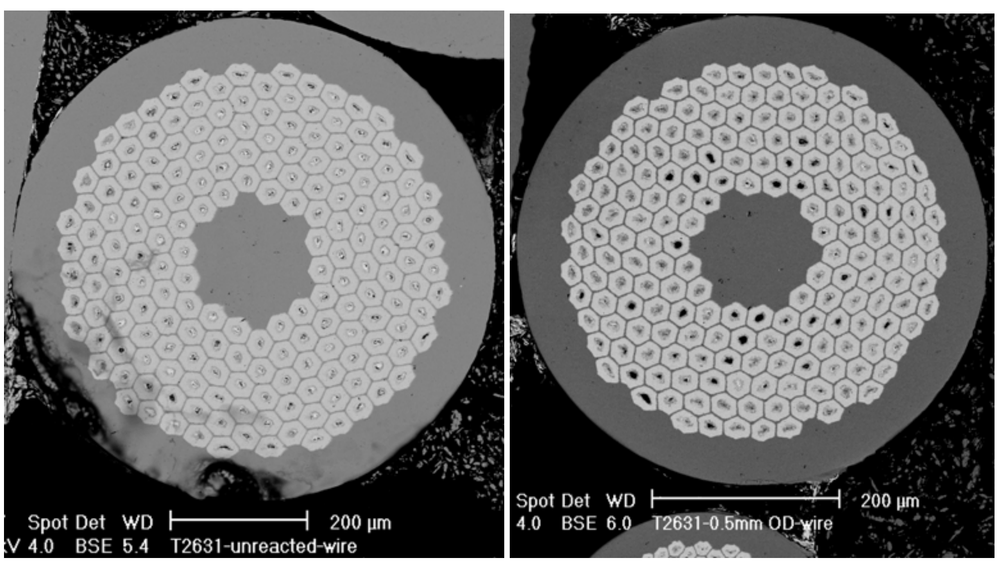


Figure 14. Subelement structure with Sn1.5at%Ti alloy in the middle.

The subelement was drawn down to restack size, hexed and then restacked into the ¾” Cu tube. The restacked strand was denoted as T2631. This strand was drawn down to 0.5 mm, 0.325 mm, and 0.25 mm without any breakage. Figure 15 shows the strands cross-sections at 0.7 mm and 0.5 mm in diameter. In the 0.7 mm, 0.5 mm, 0.325 mm, and 0.25 mm strands, their subelement size is 35 µm, 24 µm, 16 µm, and 12 µm respectively, which is similar as the subelement size of 217-subelement, 397-subelement, 919-subelement, and 1657-subelement restacked strand at 0.7 mm.



(a) 0.7 mm (b) 0.5 mm

Figure 15. T2631 strands at different diameter.

The 0.7 mm strand was heat-treated at different schedules, and OSU characterized the samples. Figure 16 shows some cross-sections after heat-treatment. Figure 17 shows its non-copper-J_c curve versus field. Its non-copper-J_c at 12 T is about 2123 A/mm^2, which is low compared to typical undoped wires. However, this is what we expected since we experienced this when we did the doping with Ti for RIT strands. The cross over to increase J_c did not occur until we reached 15T. During the Phase II we plan on getting wires tested to 15T and beyond, and compare doped and undoped wires with 1.5at%Ti in Sn. Also, looking at the reacted cross-section at 625 °C for 150 hours in Figure 16, we see there is 30-40% Nb not reacted, which will explain the relatively low J_c value, too. In Phase II, we will increase the heat-treatment duration to improve the J_c and optimize the Nb/Sn ratio in the strand.

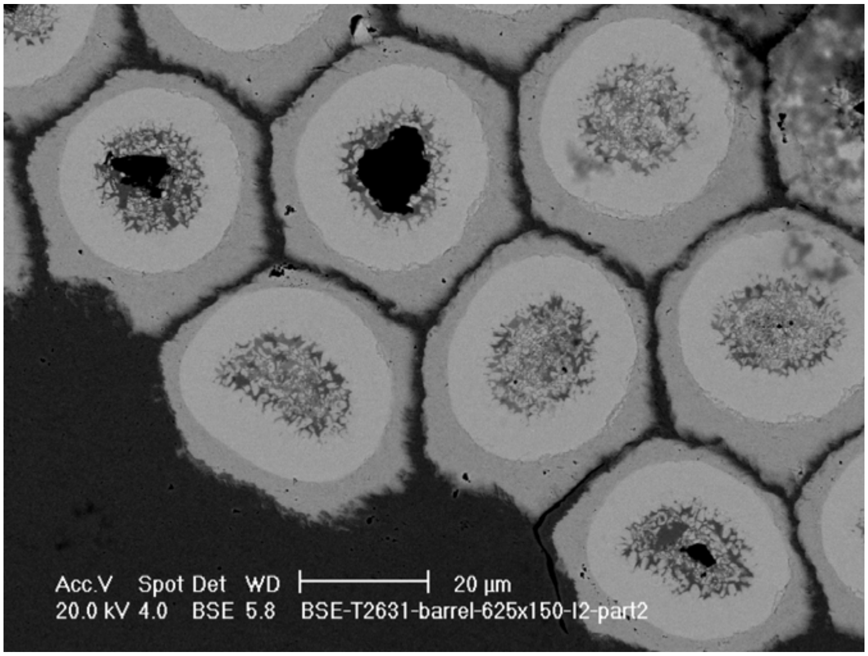


Figure 16. Cross-section of T2631 after reaction at 625 °C for 150 hr.

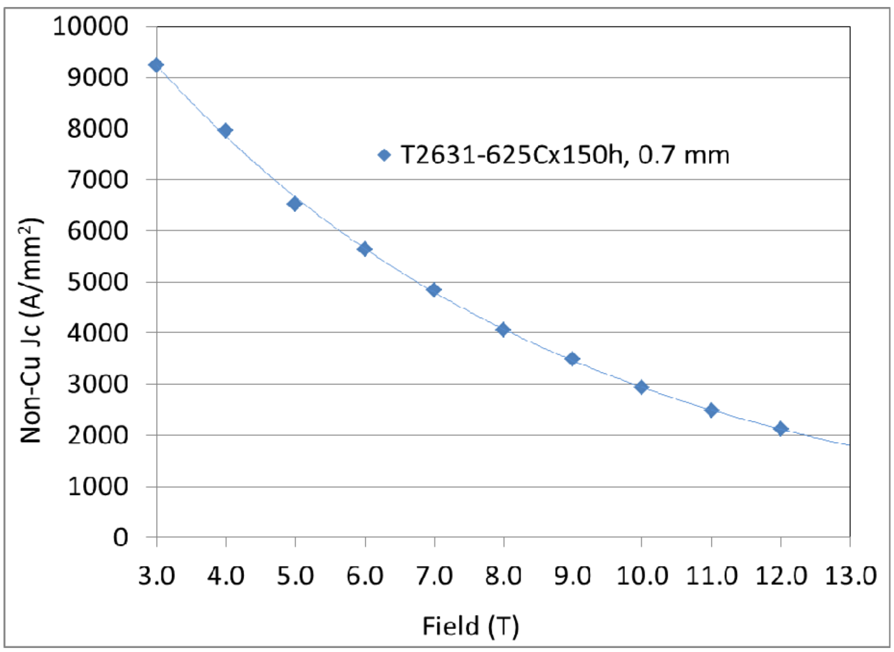


Figure 17. Non-Cu J_c with field for 0.7 mm T2631 strand.

Task 5. Characterize the Restacked Strands.

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

Electro-optical Characterization: OSU did all the SEM observation on the samples.

Superconducting Property Characterization: OSU performed the transport J_c measurements of strands up to 12 T. OSU plans on measuring various strands at high fields (15T and higher) at the High Field Magnet Lab this summer.

SUMMARY

In this Phase I, we successfully made strands with better Cu/Sn ratio to reduce the coarse Nb_3Sn grain region, thereby providing the potential of increasing the non-Cu J_c in the Phase II and scaling up to 2" billets with 331 subelements. In order to improve the strand's high field properties, we successfully doped low amount of Ti in the subelements and made a 217-subelement wire which has been drawn down to 0.7 mm without any breakage. This strand gave subelement size of 35 µm. We will scale up the Ti-doped billet to 271-subelement in 1.5" billet in this proposed Phase II. The hexagonal shaped subelements with round Nb-Sn have been developed for a 61-subelement restack. Thus the results indicated that for 217-subelement restack in a 2" billet we have the potential to draw down this type of construction without problems while maintaining a good array to react more Nb to get higher non-Cu J_c in the Phase II.

In this Phase II, we will continue working on our goal to develop a conductor capable of achieving non-Cu J_c's of about 3000 A/mm^2 at 12 T-4.2K and 1500 A/mm^2 at 15T-4.2K with filament size of <u>25~30 µm</u>.

PHASE II PROJECT

Technical Objectives

In the Phase II we have the following objectives:

1. Further optimize the Cu/Sn ratio to reduce the coarse Nb_3Sn grain region, thereby providing the potential of increasing the non-Cu J_c and scaling up to 2" billets with 331 subelements, 5-10 ft billet length that can yield strand of 7-15 km piece lengths at 0.7 mm.
2. Improve the strand's high field properties at 15T and above by doping low amount of Ti in the subelements of size 35 µm and less, and scaling to 1.5" billets with 271 subelements, 5-10 ft billets yielding 5-10 km lengths of 0.7 mm strand.
3. Use hexagonal shaped subelements with round Nb-Cu-Sn and demonstrate 217 subelements restack in a 2" billet, 5-10 ft long to react more Nb to get higher non-Cu J_c. This will yield 7-15 km piece lengths of 0.7 mm strand.

4. Supply multiple km lengths of improved Nb_3Sn tube approach strands to the National Laboratories for Rutherford cables and magnet fabrication.

The work details for this Phase II are described in the following tasks.

Task 1. Make a 217-subelement restacked strand with optimized Cu/Sn ratio and Nb/Sn ratio.
Task 2. Scale up the billet from Task 1 to make a 331-subelement in 2” OD billet with the best design. This will give a stable conductor with d_{eff} of 27 µm at 0.7 mm.
Task 3. Use the Sn1.5at%Ti and make a 217-subelement in ¾” Cu tube with more Sn to react more Nb while maintaining good RRR. This billet will be compared to T2631 from Phase I.
Task 4. Scale up the billet to 1.5” OD size with 271 subelements, and make subelements with Sn1.5at%Ti, and the best Nb/Sn ratio from Task 3.
Task 5. Scale up the billet to 2” OD size with 217 subelements, and make special subelements with hexagonal Cu around round Nb to maximize the reaction of the available Nb.
Task 6. Make a 331-subelement strand with the best design while increasing the superconductor fill factor in the strand. This will give a stable conductor with d_{eff} of 27 µm and greatly increased J_e but maintain stability at low fields.
Task 7. Characterize the restacked strands.
Task 8. Make strand available for cable and small test coil in National Labs.