Final Report**DE-FG02-07ER84860****Project Title****Improvement of the Properties of Tubular Internal-Tin Nb₃Sn****Principal Investigator****Eric Gregory****Supergenics I LLC
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Improvement of the Properties of Tubular Internal-Tin Nb₃Sn

Significance, Background Information , and Technical Approach

Significance

The overall problem is to develop an economical process that can produce material with good properties for high field magnets to be used in the future for High Energy Physics (HEP) applications.

The internal-tin (IT) process, called by some the Restacked Rod process (RRP), for making Nb₃Sn has been very successful in achieving high J_c properties in the high field region [1]. As a result it has been used effectively in several high field magnets [2]. Originally, when this material was processed to give the highest J_c it behaved unstably in the low field region and consequently did not perform as well in magnets designed for intermediate field applications [3]. In this field range, the Powder-in-Tube (PIT) material [4], that has a lower d_{eff} and a high RRR, behaved more reliably. The IT material has been improved to give better stability in the low field range [5] and consequently the process offers a considerable challenge to the PIT process for application in both types of magnets. The PIT material has two principal drawbacks – lower J_c and high cost. Work has been carried out to address these two problems [6]-[13] and Supergenics I LLC has reported, [10]-[13], on a low cost tubular process that is under development. It has fewer problems than the (IT) process in making low d_{eff} materials and is of lower cost than both the IT and PIT processes. At the present stage, the J_c 's that have been achieved are similar to those of the PIT material but are below those of the IT material.

The purpose of the work proposed here is to improve the properties of the material made by this tubular process that has been described previously [7], [10-13],

Background

Nb₃Sn material has been made by simply inserting Cu clad Sn into a Nb7.5wt.%Ta tube that, in turn, is inserted into a Cu tube and the assembly drawn down to form a sub-element. Many of these sub-elements are then inserted in another Cu tube and the restacked material again drawn down to a fine wire. All the processing is carried out cold and no Electron Beam (E.B.) welding, Hot Isostatic Pressing (HIP'ing), extrusion (either hydrostatic or conventional) or expensive powders are employed. The result is a much more flexible and economical process than the IT and PIT methods.

Several variants have also been tried. Nb tubes have been used in place of Nb7.5wt.%Ta ones and Sn2wt.%Ti has replaced the Sn in the core. The Nb and Nb7.5wt.%Ta has also been wrapped with a Ta barrier to reduce the tendency of the Sn to go into the Cu matrix but these sub-elements could not be made into restacks of more than 19-sub-elements. The low ductility of the Ta was believed to be the cause of the wire breakage and this approach requires a more ductile Ta such as that being developed by Shear Form Inc, (SFI), Bryan, TX, using Equal Channel Angular Extrusion (ECAE) [14].

The earlier past work was outlined in the proposal for this work and has been reported in papers and presentations, e.g. CEC/ICMC meeting in Chattanooga TN [12], MT 20 in Philadelphia [13] and LTSW'07 in South Tahoe CA [11]. This data will not be given in detail here. However, to remind the reviewers of the state of the Supergenics I LLC work at the time when the proposal for this work was written, we have included below the summary, given at that time, of the results of work already completed.

1. There are very few problems in making restacks with as many as 169 sub-elements in them and in drawing them down to 0.7 mm $\varnothing$ wires where the sub-elements are 33 μ m in diameter. This is true even though all the operations were carried out cold.
2. The measurements on the sub-elements show that the layer J_c 's obtained are at least equivalent to those obtained in the IT approach and stable behavior is shown down to 10T, even though the size of the Nb/Nb7.5wt.%Ta is significantly larger than that of the filaments in the IT approach [10].
3. The limitation in the non-Cu J_c in the tubular process is governed by the extent to which the Nb/Nb7.5wt.%Ta can be converted to A-15 before the Sn leaks out into the Cu matrix. The best non-Cu J_c obtained at OSU in a restack is 2173 A/mm² at 12T and 1163 A/mm² at 15T in a 55+6 restack. In a 102+25 restack where the sub-element size is 45 μ m in a 0.7 mm $\varnothing$ wire, the values obtained were 2016 A/mm² at 12T and 1102 A/mm² at 15T and the RRR was 32. Second and third samples gave essentially the same results and Brookhaven National Laboratory (BNL) measured an even higher non-Cu J_c of 2223 A/mm² at 12T. In a 162+7 restack where the sub-element size is 33 μ m in a 0.7 mm $\varnothing$ wire, the values obtained were 1819 A/mm² at 12T and 944 A/mm² at 15T. We have not had time and funds to work further on the array and heat treatment optimization but this will be done on the work proposed here.
4. The spacing to diameter ratio (s/d) of the sub-elements in the 55+6, 102+25 and 167+2 arrays are lower than in some of the other restacks and this reduces the "sausaging" and significantly increases the J_c .
5. The sub-elements on the corners of the hexagonal array are usually removed, as these are the ones experiencing maximum distortion. However the remainder of the outer row of sub-elements is also more distorted than the average. In some areas they are uneven pentagons rather than the hexagons shown by the rest of the array. Improved shimming of the outer rows of sub-elements has been tried on the 169 sub-element array but the corner sub-elements were not removed.
6. Only in the 102+25 and the 162+7 restacks have the central sub-elements been replaced by Cu. Since dynamic as well as adiabatic stability appears to be an important factor, future restacks will have reasonably sized Cu areas in the center of the restack.

An examination of the cross sections of the arrays at that time showed some of the sub-elements to be less reacted than others and this was particularly noticeable as the number of the sub-elements was reduced and their size increased.

In this work we have made significant progress in the development of the process as is illustrated in the section "Degree to which this work has Demonstrated Technical

Feasibility” below. This will give the reviewers the recent background and bring them up to date on the present state of development.

Technical Approach

The overall objective is to prove out and commercialize this new tubular Nb₃Sn with high J_c , and low d_{eff} . Supergenics I LLC is using Hyper Tech Research Inc. (HTR) as a vendor for sub-element assembly, restacking of billets, and drawing of the restacks. HTR gained the processing knowledge for tubular type Nb₃Sn, from functioning as a vendor for Supergenics I LLC and has indicated their desire to license the technology and offer Nb₃Sn wire commercially to HEP and others. HTR intends to be a commercial wire supplier and has already commercialized, and is offering for sale, magnesium diboride superconductor wire. HTR has all the equipment needed except for a large draw-bench that is required to reduce the 1.5” diameter assemblies to 0.75”. For the large draw-bench we are presently working with Luvata and they have been very helpful and cooperative. Other possibilities are Phelps Dodge in Bayway NJ and Latrobe Specialty Steel in Latrobe PA.

HTR has made a comparison between the manufacturing cost for Internal Sn (RRP type) and the tubular type with a solid Sn core. The results of this analysis were given in the proposal for a recent Phase I SBIR and they are shown in Table I below. They did not include what a company might add for additional G&A, and profit, as this is company dependent. A manufacturing labor and overhead rate of \$80/hr was assumed for both approaches.

In addition to lower material and labor costs for the tubular process, the in-process inventory cost, and in-process manufacturing costs are less. For the IT route, it can take 3-4 months to process a billet when dealing with the outside vendors, while for the tube type process, most of the operations can be carried out in house and the total time to process of a billet to final size can be as low as 3-4 weeks.

For the IT approach the following had to be considered:

Materials - Nb7.5wt%Ta rods and barrier material, Cu tubing for rods, large Cu billet for extrusion can and costs for machining it, solid Cu filaments, Cu tubing for restack and Sn for gundrilled sub-elements.

Outside services - EB welding, HIP'ing, extrusion and gundrilling of billets.

Labor and Overhead for: preparing and drawing Nb/Cu filaments and Cu filaments, cleaning can assembly of extrusion billet, drawing sub-elements to restack size (large draw bench drawing, bull block drawing), restacking of sub-elements and drawing to final size (large draw bench drawing, bull block drawing, and final fine wire drawing).

For the tubular process the following had to be considered:

Materials - Nb7.5wt%Ta tube, Cu for inner sub-element tube, Cu for outer sub-element tube, Cu for outer restack tube, Sn rod for the sub-element.

Labor and Overhead for: cleaning assembly, drawing of sub-elements, cleaning and assembly of restack billet, drawing to final size (large draw bench drawing, bull block drawing, and final fine wire drawing). The effect of these different operations on the costs, are clearly shown in Table I.

Table I

For 0.7 mm wire	\$/kg	\$/m	\$/k-Am Assume 600A, 12T (J_c 3000A/mm ²)	Based on billet Size piece lengths of
Internal Sn Cost	614	2.52	4.21	13km
Tube Type Cost	446	1.88	3.14	9km

Degree to which this work has Demonstrated Technical Feasibility

At the time when the proposal for this work was written the best properties that had been obtained with the tubular material, have been reported above and were for T 1135, a 127 (102+25) sub-element material, where the sub-element size was 45 μ m. The J_c , measured at BNL, was 2223 A/mm² in the non-Cu at 12 T. A sample of material with 169 (162+7) had been prepared with a sub-element size of 33 μ m and the best J_c value obtained was 1819 A/mm² at 12 T and 944 A/mm² at 15 T.

The Technical Objectives laid out in the proposal, were:

1. Make and test a restack with 217 (204+13) in a 0.7 mm wire where the sub-elements are 33 μ m in diameter.

2. Improve the J_c in the non-Cu as much as possible while maintaining the d_{eff} and RRR. After improving the array, the composition of the sub-element was to be changed to allow more conversion of the Nb7.5wt.%Ta.

3. Lower the d_{eff} even further by making a 271 (246+25) sub-element restack.

It was also stated that an attempt would be made to answer questions such as:

1. How high a J_c can we achieve in sub-elements that are 33 μ m or smaller in diameter?

2. What are the most important factors limiting J_c ?

3. How does J_c change as the d_{eff} is reduced?

4. How many sub-elements can be practically restacked in the small diameter tubes that have been used to date?

As will be seen from the results reported below we have met all three of the technical objectives. Some answers to the questions are also in the reported results.

Experiments to Improve the Shape of the Sub-elements in the Restack

In the proposal for the Phase I work it was pointed out that it is highly desirable to try to maintain a circular shape for the outside of the Nb7.5wt.%Ta tube after restacking. A cross section of an improved sub-element was shown and it was expected that the round O.D. would be maintained after restacking. As reported in our paper given in Chattanooga at the CEC-ICMC [12], a 127 (114+13) restack (T 1204) was made from the improved sub-elements but the tubes did not maintain their round shape after restacking. Fig. 1a shows an overall cross section at 0.7 mm diameter before reaction and Fig 1b the sub-elements at a higher magnification after reaction. This shows that they are slightly less hexagonal but more irregular than in the previous arrays and the spacing between them appears to be somewhat greater. The material was heat treated for various times at 650 °C and the best non-Cu J_c from this restack, where the sub-elements were of an

intermediate size (i.e. 35–40 μm), is shown in Fig. 7 and Table II and was 1918 A/mm² at 12 T and 1251 A/mm² at 14 T.

At this point we temporarily abandoned efforts to maintain a round configuration of the sub-element tube after restack. We may consider this approach again at a later date. It can be seen from the Figs. 1a & b that the shimming with Cu sub-elements discussed below had been used in T 1204, as the outer rows of sub-elements are somewhat hexagonal.

Table II

Sl. No.		New
Wire Name		T1204-169re-I3-d070
Description	Wire type	169 restack
	Diameter	0.7
	HT Schedule	650°C, 60h
	Sample type	ITER
Tested on		23/1/2007
I _c (A) at Fied of	10T	388
	11T	323
	12T	256
	13T	210
	14T	167
	15T	130
Non-Cu J _c (A/mm ²) at Field of	10	2907
	11	2420
	12	1918
	13	1573
	14	1251
	15	974
n-Value at Fied of	10T	25
	11T	21.4
	12T	23.9
	13T	22
	14T	21.3
	15T	21
Non-Cu Area (mm ²)		0.133478511
Non-Cu Area Fraction		0.358

Improvement in the array

It was clear from the earlier work that, when hexagonal sub-elements were packed into tubes with a simple hexagonal bore and the composite reduced to the final wire diameter, the outer row of sub-elements tended to become pentagonal instead of maintaining their hexagonal shape, Fig. 2. Initially a partial improvement was obtained by replacing the outer row of sub-elements by relatively hard hexagonal Cu elements. This had the effect of increasing the total amount of Cu in a wire of given diameter, decreasing the number of active sub-elements with each of them somewhat smaller in size than would be normal for that number, Fig. 3. It can be seen that this allowed the outside sub-elements to maintain their shape. It was not considered however, an ideal way to assemble the sub-elements in the most compact manner. In an effort to do this a Cu tube with a serrated I.D. was purchased with the aim of assembling a 217 (192+25) array. The dimensions of the tube are shown in Fig 4. This was believed to be the largest number of sub-elements that could be conveniently assembled in a 0.75" O.D. tube. (An answer to question 4)

As a third objective of this work is to make a 271 (246+25) this has to be done in a larger size tube with a similar serrated I.D. A tube with the dimensions shown in Fig. 5 was therefore purchased.

The success of producing good arrays using the customized 0.75" Ø Cu tube for 217-sub-element arrays, is best shown in Fig. 11 (T 1424) and Fig 15c (T 1505). Up to the present time only one restack has been made using the customized 1.5" Ø Cu tube. This is T1489, a 271 sub-element restack and the improvement in the array is not yet as noticeable as it is with the smaller diameter tubes.

Fig. 1. T 1204 0.7 mm dia. (a) Unreacted, (b) 60 h at 650 °C. Sub-element size 35-40 µm.

Fig. 2. Cross-section of T1135 (102+25) showing pentagonal sub-elements in the outer rows. Sub-element size 45 μm .

Fig.3. Cross section T 1319 (114+13) with a row of hexagonal Cu elements in the outside row. a. Unreacted b. after 100 h at 650 $^{\circ}\text{C}$. Sub-element size 35-40 μm .

Fig.4. 0.75" customized Cu tube for 217 (192+25) restack

Fig. 5. 1.5" customized Cu tube for 271 (246+25) restack

Change in the percentage of Cu inside the Nb7.5wt.%Ta tube

It was also mentioned in the proposal that there appeared to be a possibility that in some of the assemblies the Cu tube used to surround the Sn core did not reduce evenly as the wire was drawn down. In some cases it appeared to actually break up and possibly caused wire breakage. The wall thickness of the tube used for this purpose was therefore increased from 0.010" to 0.020" and the latter was what was used for the T 1319 restack. This was otherwise similar to T 1204 although no particular effort was made to maintain the O.D. of the Nb7.5wt.%Ta tube in a circular shape. The change in the Cu tube had the effect of altering the composition of the sub-element as shown in Table III. T1025 is an earlier sub-element with a 0.015 " wall thickness Cu tube and T 1302 the sub-element that was used for the T 1319 restack.

A cross section of this T 1319 material at 0.7 mm diameter is shown at a low magnification in Fig. 3a and at a higher magnification after 100 h at 650 °C, Fig. 3b. The J_c at 12 T was 1766 A/mm² and at 14 T 1059 A/mm². Another test was made with a heat

treatment time of 120 h and the non-Cu J_c results at 12 T were 1933 A/mm² and at 14 T 1150 A/mm², as shown in Fig. 7 and Table IV.

A.C. loss experiments were carried out on the T 1204 material that had a sub-element size of 35-40 μ m, to measure its d_{eff} . Two different samples and two different pieces of measuring equipment were used in this work. Sample S4 consisted of a series of short straight lengths heat treated for 60 h at 650 °C and measured over a field range of $\pm$ 14 T. The value of d_{eff} obtained was 33 μ m and the magnetization curve is shown in Fig. 6. Sample A4 consisted of a relatively long coil heat treated for 72 h at 650 °C and measured over a field range of $\pm$ 8 T. The value of d_{eff} obtained was 51 μ m.

Considering the many variables involved, the results are relatively close to the measured sub-element size. More details on the procedures used for a.c. loss measurement are given in a paper by Sumption et al in Cryogenics [15].

In order to further reduce the sub-element size a 156+13 sub-element restack (T 1339) was made with the new composition of the sub-element and a cross section of this material at 0.7 mm diameter is shown at low magnification in Fig. 8. The sub-element size is 33 μ m. The J_c properties are given in Table V. A sample of this material at 0.7 mm $\varnothing$ was sent to BNL and a J_c of 2162 A/mm² in the non-Cu at 12T was obtained. This result was in complete agreement with the best result from OSU (Table V and Fig. 9). This is an improvement over the previously reported 33 μ m $\varnothing$ sub-element material that gave 1819 A/mm² in the non-Cu at 12T. (Second Technical Objective)

In view of the success with the 0.7 mm $\varnothing$ properties of T 1339, it was drawn down to 0.52 mm $\varnothing$ where the sub-element size was $\sim$ 25 μ m (Figs. 10a & b). This was heat treated for 40 h at both 630 °C and 650 °C and the non-Cu J_c s at 12 T obtained were 1817 and 1714 A/mm² respectively. As can be seen below this approach of drawing to a smaller size than 0.7 mm, i.e. 0.42 mm, on the newer assemblies, has shown that even material with 18 μ m $\varnothing$ sub-elements gave J_c properties >2000 A/mm² at 12 T. This addresses the third question listed above. There is however a general feeling that to get the best answer, a 0.7 mm $\varnothing$ wire with sub-element number of greater than 400, will have to be made.

As mentioned above a 0.75" diameter Cu tube with a serrated I.D. designed to take 217 hexagonal sub-elements was obtained and one of the first restacks made with this was designated as T 1424. A cross section of which at 0.7 mm $\varnothing$ is shown in Figs. 11a & 11b. This material was tested at both OSU and BNL at 0.7 mm $\varnothing$ and 1 mm $\varnothing$. The results are shown in Fig. 12 and Table VI.

These show the successful achievement of the First Technical Objective.

Fig. 13 and Table VI give the J_c data obtained at BNL on different samples of tubular material tested up to that time and Table VII shows the stable current density, J_s and the RRR's. Fig. 14 shows a plot of J_s against sub-element size for materials with low

Table III. Composition of Sub-elements in volume %

Strand	Sn	Nb	Cu inside	Nb7.5wt.%/Sn
T1025	23.52	72.23	4.25	3.07
T1302	24.19	67.32	8.49	2.78

Table IV

Sl. No.		2	2
Wire Name		T1319-169re-I4-d070	T1319-169re-I5-d070
Description	Wire type	Regular HT	Regular HT
	Diameter	0.7	0.7
	HT Schedule	650°C, 100h	650°C, 120h
	Sample type	ITER	ITER
Tested on		2/8/11	4/8/11
I _c (A) at Field of	10T	376	399
	11T	307.4	318.28
	12T	236.2	258.63
	13T	182	201.5
	14T	141.69	153.83
	15T		
Non-Cu J _c (A/mm ²) at Field of	10	2811	2982
	11	2298	2379
	12	1766	1933
	13	1360	1506
	14	1059	1150
	15	-	-
n-Value at Field of	10T	Q	Q
	11T	66.7	35.8
	12T	24.8	47.8
	13T	18.3	37.8
	14T	32.5	34.7
	15T	-	-
Non-Cu Area (mm ²)		0.133783499	0.133783499
μ		0.359	0.359

Fig. 6. Magnetization curve ± 14 T of T 1204 sample S4 after 60 h at 650 °C.

Fig.7. J_c vs. B for T1204 and T1319

Fig. 8. Cross section of T 1339 at 0.7 mm unreacted.

Fig. 9. J_c vs. B for T1339

Table V

Sl. No.		T1339-217re-I1-d070	T1339-217re-I4-d070	T1339-217re-I6-d070
Wire Name				
Description	Wire type	217 Restack	217re	217re
	Diameter	0.7	0.7	0.7
	HT Schedule	650°C, 80h	650°C, 80h	650°C, 90h
	Sample type	ITER	ITER	ITER
Tested on		8/9/11	10/6/11	10/11/11
I _c (A) at Field of	10T	510	475	483
	11T	410	382	397.46
	12T	340.7	310	321.21
	13T	273.9	246	256.65
	14T	213.6	188	199
	15T			
Non-Cu J _c (A/mm ²) at Field of	10	3239	3016	3067
	11	2604	2426	2524
	12	2164	1969	2040
	13	1739	1562	1630
	14	1356	1194	1264
	15	-	-	-
n-Value at Field of	10T	Q	Q	27
	11T	Q	Q	33.9
	12T	Q	Q	35
	13T	Q	Q	36.2
	14T	Q	Q	32
	15T	-	-	-
Non-Cu Area (mm ²)		0.15747571	0.15747571	0.15747571
Non-Cu Area Fraction		0.4094	0.4094	0.4094
A15 Layer Area Fraction

Fig. 10. T 1339 at 0.52 mm Ø Unreacted. a. Low magnification. b. High magnification

RRR's showing a clear increase in J_s as the size is lowered. Several efforts have been made to test T 1424 material at 0.42 mm Ø (this also has a Nb7.5wt.%Ta/Sn of 2.5) at OSU to get an indication of what the J_s would be with a sub-element size of $\sim 20 \mu\text{m}$. Fig.14 predicts a high value even though the RRR will be low. Unfortunately OSU has had significant problems testing this fine wire and BNL successfully tested it but the J_c at 12 T was only 1358 A/mm^2 and because of this the J_s of 6951 A/mm^2 was of less significance.

The 0.42 mm Ø material after the 60h at 650°C heat treatment was examined metallographically and the results are shown in Figs. 15a & 15b. It is obvious from these photos that the Sn has leaked out into the matrix and is reacting with the Nb on the outside of most of the sub-elements. An EDS measurement was made on the middle of the fine-grained A-15 array and the results are shown in Table VIII. The relatively high value of 23.54 at.% for Sn suggests relatively good stoichiometry.

The material T 1135 with a sub-element size of $45 \mu\text{m}$ has a much higher J_s and RRR than all the other samples measured up to that time, presumably due to less matrix contamination, (Table VII). The Nb7.5wt.%Ta/Sn ratio of T 1135 was 3 and that of the materials made later was 2.5. The first restack with 271 sub-elements, T 1489, was made with a Nb7.5wt.%Ta/Sn ratio of 3 as was another 217 sub-element restack, T 1505, shown at 0.7mm in Fig. 15c where it appears to have a particularly uniform array. This T 1505 material was heat treated for 100 h & 120 h but has yet has not been tested. The T1489 material at 0.7 mm Ø was heat treated for 100 h at 650°C and the same material drawn to 0.42 mm Ø and heat treated for 60 h at 650°C . Both samples were sent to BNL for testing. The 0.42 mm Ø material had a J_c of 2038 A/mm^2 at 12T and a J_s of $12,357 \text{ A/mm}^2$. The 0.7 mm Ø material had a similar J_c of 2030 A/mm^2 at 12T and a J_s of 4170 A/mm^2 . The smaller diameter wire is impractical for magnet winders but its performance helps us to determine what to expect from 469 sub-element material. The problems of getting data from 0.42 mm Ø wire at OSU point out the necessity of making material where the 469 sub-element material has a sub-element size of $22 \mu\text{m}$ in a 0.7 mm Ø wire.

The results with the 271 sub-element material made in a 1.5 “ Ø Cu tube suggest that the likelihood of success to be quite high.

a.
b.
Fig. 11a & b. T 1424 at 0.7 mm after 80 h at 650 °C.

Fig. 12. J_c vs. B for T1424

Fig. 13. Jc vs. B results from BNL Tests

Table VI

	BNL T1403- 0.7mm 650Cx150h	BNL T1135- 0.69mm 650Cx72h	BNL T1339- 0.7mm 650Cx90h	BNL T1424- 1.0mm 650Cx130h	BNL T1424- 0.7mm 650Cx90h	BNL T1424- 0.7mm 650Cx72h	OSU T1424- 0.7mm 650Cx72h
14.00							1272
13.0							1543
12.0	2079	2223	2162	1934	1945	1888	1899
11.5	2293	2662	2376	2143	2153	2094	
11.5	2293	2429	2376	2143	2153	2094	
11.5	2293	2414	2376	2143	2153	2094	
11.5	2293	2436	2376	2143	2153	2094	
11.0	2523	2464	2606	2370	2377	2317	2300
10.5	2773	2911	2855	2615	2620	2558	
10.0	3043	3226	3124	2882	2883	2820	
9.5	3336	3493	3416	3171	3169	3105	
9.0	3654	3816	3733	3486	3480	3415	
8.0	4380	4512	4454	4206	4191	4123	
7.0	5256	5397		5075	5048	4979

Fig.14. Js vs. Sub-element size

Table VII

	Deff (sub-element size)	Is	Js	Jc at 12 T	RRR
T1403	66	250	1384	2079	3.4
T1339	30	375	2378	2162	3.2
T1424-0.7mm 80 h	30	450	2491	1888	3.3
T1424-0.7mm 90 h	30	425	2352	1945	2.3
T1424-1mm	53	575	1559	1934	3.8
T1135 72 h	45	775	4546	2225	73
T1135 48 h	45	1075	6305	1391	244

Effects of increasing the amount of Cu in the core.

It was suggested by OSU that increasing the Cu percentage in the core might increase the amount of fine-grained Nb_3Sn relative to the coarse. We therefore made another restack, T 1449, similar to T1424 from a sub-element that had the Sn enclosed in a Cu tube with a wall thickness of 0.035" instead of 0.020". A cross section of the material at 0.7 mm $\varnothing$ is shown in Figs 16a & b. It was heat treated at 650 °C for 80 & 90 h. The non-Cu 12 T J_c was 1576 A/mm² & the J_q 1632 A/mm² after 80 h and after 90 h it showed no improvement. It was decided at this point to concentrate on a Cu tube of 0.020" thickness.

Table VIII

Element	Atomic %
Nb	74.22
Sn	23.54
Cu	0.86
Ta	1.38

Fig. 15a & b. 0.42 mm Ø T 1424 after 60 h at 650 °C.

Fig. 15c. 0.7 mm Ø T1505, 120 h at 650 °C

Figs. 16a & b. T 1449 at 0.7 mm Unreacted.

The successful achievement of the third objective

In order to get an indication of the effect of lowering d_{eff} without having to make restacks with very large numbers of sub-elements, we have taken the available materials and drawn them down to 0.42 mm Ø. This we have done using the 217 sub-element material made in 0.75" OD Cu tubes but we have had some problems handling and testing this fine wire at OSU. We had already proposed making restacks containing more sub-elements and this meant that we had to scale up to restacking in a 1.5" Ø Cu tube. In this work we have taken the important first step by making a 271 sub-element restack in such a tube.

This we have done successfully and the result is shown in Figs. 17a & b. These show the T1489 restack at 0.7 mm Ø after 90 h at 650 °C. A 4 ft. long restack in a 1.5" Ø Cu tube was assembled at HTR and drawn down to 0.7" Ø at Luvata, Waterbury. They also drew down a length to 1 mm Ø showing that this approach could make 30 μm Ø sub-element material for ITER barrel samples of 0.7 mm Ø. HTR has subsequently drawn this material to 0.42 mm Ø, (Fig.17c), and 0.27 mm Ø, (Fig. 17d), wire. The 0.7 mm Ø T 1489 wire was heat treated for 100 h at 650 °C and the 0.42 mm Ø for 60 h at 650 °C. The cross sections after reaction are shown in Figs. 18 a, b, c & d. The results from BNL are shown compared with those of T 1135 in Table IX and Fig. 19. It is important to note that these are however the first results from this billet and it is expected that the properties will be increased somewhat by optimization of heat treatment. At BNL's request we have sent them an additional 10 m of 0.42 mm Ø and 10 m of 0.7 mm Ø T 1489 wire for such a heat treatment investigation.

The above results show that we have successfully achieved the third objective of this work.

Fig. 17a & b T 1489, 271-sub-element restack at 0.7 mm after 90 h at 650 °C

Fig.17c. 0.42 mm Ø T1489, unreacted.

Fig.17d. 0.27 mm Ø T1489, unreacted

The additional material from Luvata, some at 0.7" Ø, some at 0.23" Ø and the 1mm Ø sample were returned to HTR and the latter has subsequently been drawn down to 0.7 mm Ø and even smaller as mentioned above.

As we have already pointed out, this success was particularly important as it indicated that we have a very good chance of making 331, 397 and 469 sub-element restacks from 1.5" Ø Cu tubes. In this way we will be able to make 22 μm Ø sub-elements in a 0.7 mm Ø wire. Wire of this diameter will be of much more practical value than the smaller diameter wire.

Summary of the work.

The above has shown that we have met all the objectives of the work laid out in the proposal.

1. We have made and tested a 217 (192+25) sub-element restack.
2. The J_c in a material with 33 μ m sub-elements has been increased from 1819 A/mm² at 12 T to 2162 A/mm² at 12 T in T 1339.
3. We have made a 271 (246+25) sub-element restack, T 1489, using a large (1.5") $\varnothing$ restack and successfully drawn it down to 0.27 mm $\varnothing$ wire where the sub-element size is $\sim$ 12 μ m. At 0.7 mm $\varnothing$ it gave a J_c of 2030 A/mm² at 12 T and at 0.42 mm a J_c of 2038 A/mm² at 12 T. (The sub-element size of this material was 18 μ m), These are the first results on billet T 1489 and it is expected that they will be improved by optimization of the heat treatment.

Figs. 18 a & b, T 1489 0.7 mm $\varnothing$ after 650 $^{\circ}$ C for 100 h

Figs. 18c & d, T 1489 0.42 mm $\varnothing$ after 650 $^{\circ}$ C for 60 h.

Answers to the questions posed in the proposal.

The most important factor limiting J_c is converting as much as possible of the Nb7.5wt.%Ta before the matrix becomes contaminated with Sn. There are indications however that as the d_{eff} is reduced J_s increases despite matrix contamination (Fig. 14).

J_c does not appear to deteriorate significantly as d_{eff} decreases in the range presently tested. We have obtained 2038 A/mm² in material with 18 μ m $\varnothing$ sub-elements. Composition and heat treatment appear to be more important variables.

We are changing the Nb7.5wt.%Ta/Sn ratio in an effort to raise the J_c above 2222 A/mm² at 12 T, the best value obtained on T 1135 a 45 μ m $\varnothing$ sub-element material. Up to the present we have only examined two Nb7.5wt.%Ta/Sn ratios, 2.5/1 and 3.0/1 and the heat treatment conditions have not been varied significantly.

The practical number of sub-elements that can be stacked in a 0.75" $\varnothing$ Cu tube is 217 (answer to question 4), but we have successfully processed 271 in a 1.5" $\varnothing$ Cu tube and drawn the product down to a diameter of 0.27 mm suggesting that there should be very little trouble stacking 469 (444+25) sub-elements in this size of tube and drawing the product to 0.7 mm. This would give 0.22 μ m $\varnothing$ sub-elements in a 0.7 mm wire. Such material should have a very reasonable J_s even if it has a low RRR.

Table IX
First results on T 1489 compared with T 1135

Field (T)	T1489-0.42mm 650C 60h	T1489-0.7mm 650C 100h	T1135-0.69mm 650Cx72h
12	2038	2030	2223
11.5	2252	2232	2462
11.5	2252	2232	2429
11.5	2252	2232	2414
11.5	2252	2232	2436
11	2482	2451	2464
10.5	2731	2687	2911
10	3001	2943	3226
9	3613	3521	3816
8	4341	4207	4512
7	5218	5033	5397
6	6297	6047	
5	7662	7328	
4	9464		
3	12018		
2	16116		
I_s	800	750	775
J_s	12357	4170	4546
RRR			73
Deffective	18 μ m	31 μ m	45 μ m

Fig 19. J_c vs. B for T1489 compared with T 1135

T 1135 material that has given the highest J_c at 12 T so far, gave much higher values of RRR and J_s than much of the subsequently processed material although the sub-elements were large (~45 μm). The reason for this was presumably that the Sn entered the matrix to a greater extent in the materials that had Nb7.5wt.%Ta/Sn ratios of 2.5. The equivalent value for T 1135 was 3.0. We recently changed the ratio back to 3 in the latest restacks, T 1489 (a 271 sub-element restack) and T 1505 (a 217 sub-element restack).

The indications from this work are that fine sub-elements improved overall performance. However achieving these by drawing the wire down to a very small size had several disadvantages and wire diameters less than 0.7 mm are generally looked upon unfavorably by the magnet designers.

In an effort to answer more definitively the first three questions outlined in the proposal (listed at the beginning of the above section entitled “Degree to which this work has Demonstrated Technical Feasibility”) we have suggested an increase in the number of restack sub-elements from 271 to 469 and have submitted another proposal to do this. As has been repeatedly stated, a restack of 469, the sub-element size will be ~22 μm at a wire diameter of 0.7 mm. This is an ambitious objective and to achieve it we will have to fix most of the other variables at the best values known at the time that this work is started and concentrate on increasing the sub-element number only.

In this work we attempted to obtain more information to answer the questions by simply drawing down the 217 sub-element and the 271 sub-element restacks to 0.42 mm Ø and 0.27 mm Ø.

Figs. 15 a & b. have shown the 217 sub-element material at 0.42 mm. An EDS measurement taken at the center of the A-15 array gave an atomic % of Sn as 23.54, showing that the stoichiometry of the A-15 is fairly close to ideal.

We have however had problems testing this 0.42 mm Ø material at OSU and the J_c value obtained at BNL on T 1424 at 0.42 mm Ø was low.

Figs. 17a, b, c & d have shown the 271 sub-element restack, T1489, at various diameters and the 0.7 mm Ø sample after a 90 h at 650°C heat treatment.

In the work on 0.7mm Ø strands the highest non-Cu 12T J_c was 2223 A/mm² achieved with a 127 sub-element restack, T1135, where the sub-element size was 45 µm and the J_s was 4546 A/mm² and the RRR 73. The Nb7.5wt.%Ta/Sn ratio was ~3.0, and a Cu tube with a 0.020" thick wall tube was used.

For most of the succeeding work the Nb7.5wt.%Ta /Sn ratio was closer to 2.5 and the increased Sn tended to break through the tubes, contaminate the matrix and cause the 0.7 mm samples to quench prematurely. As mentioned above, the wire was drawn to 0.42 mm, the sub-elements became ~20 µm in diameter, and the material was heat treated for 60 h at 650 °C in an effort to determine its J_c and J_s . Unfortunately the problems of testing this fine wire at OSU were such that we are considering an alternative approach. We will test at a diameter of 0.6 mm that is believed to be a readily testable size and to decrease the sub-element size by adding extra Cu to the out side of the wire. If this wire can be drawn down without too much breakage and "sausaging" it will enabled us to decrease the sub-element size to ~22 µm without changing the number of sub-elements in the restack from the 271 that we have presently achieved. This may be a superior way of examining the effects of d_{eff} , rather than drawing the overall wire to a much smaller size. One of the first attempts is shown in Figs. 20a and b where the sub-elements are ~22 µm and the wire diameter is 0.7 mm. The array is very uniform suggesting that this is a good way to observe the effects of decreasing the d_{eff} without having to increase the number of sub-elements.

Figs. 20a & b. T1489 that has had an additional amount of Cu and was then drawn to 0.7 mm Ø. This gives a very uniform filament array of sub-elements that are ~22 µm in diameter

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