METHODS OF IMPROVING INTERNAL-TIN Nb₃Sn FOR FUSION APPLICATIONS

PHASE II - FINAL REPORT

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PROJECT SUMMARY

Title of Project and Technical Abstract
(Limit to Space Provided)

Methods of Improving Internal-Tin Nb₃Sn for Fusion Applications

The overall objective of this work is to provide the TPX / ITER programs and similar projects with an improved, reliable and economical high field Nb₃Sn multifilamentary conductor strand made by the internal-tin process. An effort will also be made to determine the reasons for the property changes taking place after various heat treatment cycles in an effort to develop optimized heat treatments for the various applications.

While strand has been made which meets the HP-1 specifications, the margins by which it does so are small and there is a definite and urgent need to widen them. The phase I work has shown that the addition of Ge to the matrix of a Tubular Tin Source (TTS) conductor improves the J_c throughout the field range (8T – 12T). Since this type of conductor is not suitable for TPX / ITER strand, the Phase II work will be to prove that the matrix material containing Ge can be used in tin-core strands, which are used for these fusion applications.

Cost reduction is also an important factor in both these projects and we will work on scaling up the size of the stabilizer billets to 203 mm (8.0") diameter from the present 152 mm (6") diameter size in an effort to reduce the fabrication costs.

Chromium plating is presently used on strand for both these programs to prevent sintering and to reduce transconductance. Many questions have arisen from the use of this material and a dual barrier approach was used in Phase I to show that the RRR of Cr-plated material can be made relatively independent of heat treatment. It is proposed that in Phase II this approach will be modified by removing the copper and techniques developed to produce a tantalum surface.

During the Phase I work it has been determined that the losses exhibited by the wire are highly dependent on the ramp rate of the heating cycle and also on whether or not this cycle includes holds at intermediate temperatures. In phase II an extensive investigation will be carried out to find an explanation of these effects. This work will include diffusion studies and a determination of which phases are present at any stage of the heat treatment. How the distribution of the porosity and the extent of the bridging are affected, will also be explored.

Results / Potential Commercial Applications of the Research

This work has resulted in the development of a more reliable and economical high field material for ITER and TPX as well as LDX and KSTAR programs. Also the material will exist for use in high energy physics applications. In the commercial area, the improved material resulted from this work will be available for use in high field laboratory magnets.

Enclosed is a list of the technical publications by key personnel, which has resulted from the work carried out under the subject grant. Copies of all the publications related to this project have also been attached here as a replacement of the final report.

List of publications by key personnel under this project

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Development of Nb₃Sn Conductors for Fusion and High Energy Physics

Taeyoung Pyon and Hem Kanithi

Abstract—In the recent years internal-tin Nb₃Sn conductors have been developed to a stage where the critical current densities obtainable are triple those exhibited by the bronze processed material. Outokumpu Advanced Superconductors has been producing a commercial quantity of Nb₃Sn wires mainly using the internal-tin process. Nb₃Sn conductors, regardless of the method used in their fabrication, have to be made of different designs depending on their specific application. This paper describes design parameters of internal-tin Nb₃Sn conductors, which have been developed and made recently for use in fusion and high energy physics in particular. Their properties including the current carrying capacity and magnetization are given. The trade-offs between the critical current densities, AC losses, flux jumps and stability, and how the design parameters affect these properties are also discussed.

Index Terms—AC losses, critical current densities, flux jumps, fusion, high energy physics, Nb₃Sn

I. INTRODUCTION

RECENT efforts and development work to improve the properties of the multifilamentary Nb₃Sn superconductors has been highly focused on the achievement of the maximum current carrying capacity as high as possible. Since the concept of the internal-tin process was first adapted in U.S. from the Japanese group, Hashimoto [1] in the early 80's, Outokumpu Advanced Superconductors (OKAS), formerly IGC Advanced Superconductors, continues their effort to develop the high performance Nb₃Sn mostly through the internal-tin process.

From the results of the series of recent success on the testing of both the ITER Central Solenoid Model Coil (CSMC) and the Levitated Dipole Experiment (LDX) F-Coil by MIT and Columbia University [2,3], it has evidently shown that the internal-tin is the most viable material commercially available in a production quantity for the large scaled magnets [4]. Due to the significant participation and contribution to the ITER program for the conductor procurement, OKAS has also been supplying nearly 5 tonnes of the advanced quality internal-tin conductor to the Korea Superconducting Tokamak Advanced Research (KSTAR) program for fusion application [5]. This

device is designed to accommodate much more aggressive specifications for the conductor than those required for the CSMC. Because of its highly demanding requirements it would be very difficult to make such conductor with an approach other than internal-tin process. Material with similar strand specifications has also been produced for the European fusion community, Centre de Recherches en Physique des Plasmas (CRPP), in their preparation for an ITER Full Size Prototype Conductor. Once the full size of conductor is fabricated in the final form, it will then be tested in the SULTAN test facility.

The conductor requirements for the High Energy Physics (HEP) programs are much more difficult and still quite challenging to achieve than those for the fusion application. The main goal of this is to obtain the highest possible critical current densities in the non-copper area. The technical and cost performance goals for the conductor required for the devices such as the Very Large Hadron Collider (VLHC) in the HEP conductor development program have been reemphasized in more specific details [6]. In order to have a significant increase in the current densities, it is practically inevitable for the filaments to agglomerate after reaction. However, the subelement bundles still need to be separated from each other to reduce the flux jumps and the effective filament size on the reacted strand. Several approaches with different fabrication methods, including the Internal Tin (IT), Modified Jelly Roll (MJR) and Powder-in-Tube (PIT) processes are currently being utilized in this area to explore the feasibility of the VLHC goals. In this paper, the recent progress in developing the high current Nb₃Sn at OKAS through the internal-tin process will be discussed.

II. ADVANCES IN FUSION CONDUCTOR

A. Strand Requirements

The strand requirements for the KSTAR program are very challenging for both the critical current and the hysteresis loss compared to those made for the ITER. Therefore the serious design changes of the conductor were necessary to achieve the new requirements. Several tonnage of the massive production of this material has then led to the request from the CRPP group for the strand with even slightly further advanced specifications. Table 1 shows the strand requirements for the conductors developed for those programs mentioned above.

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B. Improvements in Properties

A key issue in the development of the material needed for the KSTAR and CRPP has been the significant reduction of the AC losses without diminishing the current densities. In the conventional design of the internal-tin wire, the barrier was extruded with the stabilizing copper, and then the core material was stacked together into it. The barrier was made out of the combined double layers of niobium and tantalum, where the tantalum is located inside and backed outside by the niobium to improve the ductility.

First attempt was made on the modification of the diffusion barrier. Relatively thick double barrier has been replaced with a single tantalum wrap in the restacking assembly stage. In this way the extrusion process of stabilizer was finally eliminated to be more cost effective and the fabrication process has also been further simplified. As a result of this change the hysteresis loss of the reacted wire was reduced as much as 15% [7] due to the absence of the niobium in the barrier.

In addition to this modification, the titanium was doped into the tin to enhance the properties at the higher field region. This approach resulted a further reduction of the AC losses to a certain extent. This contribution is believed to be a part of the result from the grain refinement during the reaction heat treatment. In fact there are still some groups of filaments bridged together after reaction, although the filament array has been designed in such way that the spacing is maintained to keep them separate. Another design change was made to avoid this undesirable outcome by rearranging the spacing between the filaments more effectively so that the amount of the bridging can be kept as a minimum. The cross section of the conductor made for the CRPP is shown in Fig.1 above. This material has been made with very similar approach as used in the conductor for the KSTAR program.

The non-Cu J_c for the material produced for the KSTAR were obtained in the range between 750 and 1000, with average of 874 A/mm² at 12T and 4.2K. Hysteresis loss, however, of this material was marginal and measured values were found from as low as 150 and up to 350 kJ/m³. The piece lengths on this wire have been exceedingly improved recently by employing a series of changes in the fabrication process.

Preliminary data obtained from the prototype sample wire

TABLE I
COMPARISON IN THE STRAND SPECIFICATIONS

Physical Parameters	ITER CSMC	KSTAR	CRPP
Strand Diameter (mm)	0.81	0.78	0.70
Non-Cu J_c @ 12T, 4.2K (A/mm ²)	> 650	> 750	> 800
n-value	> 20	> 20	---
Non-Cu Q_h @ $\pm 3T$ cycle (kJ/m ³)	< 600	< 250	< 300
Cu / non-Cu Ratio	1.50	1.50	0.80
RRR, 273K/20K	> 100	> 100	> 100
Chrome Thickness (μ m)	2.0 ~ 2.5	1 $\pm$ 0.5	1.0 ~ 1.5
Twist Pitch (mm)	10	13	10

Fig. 1. Cross-sectional view of the core section of 0.7 mm diameter wire produced for an ITER full size prototype conductor.

prepared prior to the production of the conductor for the CRPP, has shown that they are essentially not much different from the ones observed in the conductor made for the KSTAR. The heat treatment condition given was 185°C for 100 hrs, 460°C for 144 hrs, 575°C for 200 hrs followed by 240 hrs at 650°C. The trace of the progress on the properties of the internal-tin manufactured for fusion application is illustrated in Fig.2.

III. INTERNAL-TIN FOR HIGH ENERGY PHYSICS

In the previous work done at OKAS under the first year R&D program with Lawrence Berkeley National Laboratory (LBNL), the achievement of the non-Cu J_c of 2578 A/mm² at 12T was reported [8]. This encouraging result has been the main driving force to continue to pursue the technical goal of the HEP conductor development program.

A. Billet Design Optimization

The subelement used in the material mentioned above was prepared with the Wah Chang (WC) Nb, 20/80 Cu to Nb local area ratio (LAR) and stacked into 179mm diameter billet. The extruded subelement rod with the tin in the core was drawn and restacked into the Ta barrier and stabilizer Cu tubes. It was then proposed, for the next stage of the program, a larger subelement billet needs to be fabricated to confirm the previous work carried out in the early stage. Some of the variables were expected in this task based on the limited raw material availability and processing parameters. Nb mono ingot from Reference Metals Company (RMC) was extruded and drawn to 11.7mm diameter rod. After the full annealing heat treatment the rods were clad with 20/80 Cu tube and drawn to the stacking size of 3.28mm hex, then straightened and cut to the length. The billet was assembled into 205mm in

Fig. 2. Critical current densities versus hysteresis losses in the non-Cu are of the conductors manufactured for fusion application.

diameter and extruded with the same parameters and the conditions as used in the previous set of billets made for the early year program.

After machining the outside Cu shell and drilling the central hole for tin insertion, the tube was annealed at 810°C to soften the filaments, prior to the drawing process. Problem, however, was encountered in the early stage of the bench drawing. A pair of cracking along the rod has occurred starting from the filamentary region and propagating through the Cu jacket. A series of this type of cracking pattern has already been observed from the material made before, and it tends to happen on the material with relatively hard monofilaments. The indication of the cracking problem in this rod is shown in Fig.3. Although the rod was clad with Cu tube right after a few draw to stop the progress of cracking, it continued to develop as drawn.

After an extensive investigation focusing on the issue

Fig. 3. Cross-section of partially drawn subelement rod, showing the indication of crack in the filament region.

associated with the cracking based on the information available, a decision was made to rebuild another billet. The key change was made in the extrusion parameters, which is believed to be improving the material flow during the extrusion. Annealed Nb rod from WC has been clad with the 20/80 LAR Cu tube and processed to assemble a 205mm diameter billet. This material is now in the process of gun drilling for the tin.

B. Intrinsic Properties

Once the above objectives have been achieved, the next step would be to approach the limit to which the properties of structures of this configuration of material can be improved. In an effort to increase the intrinsic J_c within the filaments, the replacement of Nb with the Nb1wt.%Zr has been explored. It has been reported that Zr addition can boost up the J_c significantly in the low and mid range of the magnetic field. For the high field performance Ti is still being utilized to maintain the improvement of H_{c2} , although it is not yet certain about how both addition of Zr and Ti will affect the superconducting properties as well as the fabrication process.

A relatively small amount of work has been done on the use of Nb1wt.%Zr alloy in the multifilamentary material for Nb₃Sn. There has been however, a considerable amount of work done by General Electric (GE) R&D Center using this alloy in tape material [9].

Parts for the billet with Nb1wt.%Zr were prepared with basically the same configuration of the design employed in the above work. Due to the problems related with the work hardening, which are similar to the ones observed in the earlier material, the extrusion process has not been successful on this material. The second trial billet with the modified extrusion parameters did not even go through well. It is felt that this

Fig. 4. Experimental stacked material composed of the distributed Nb1wt.%Zr and SnTi rods with pure Ta diffusion barrier.

might have been resulted from the significant difference remaining in the residual flow stress between the Nb1wt.%Zr and the Cu at the extrusion temperature.

While the investigation is carried out to better understand this material, a small experimental stacked material has been prepared to explore the feasibility of drawing on this alloy, by eliminating the extrusion process. The trace of work hardening of this material will also be monitored through the drawing process. The overall cross section of this restack is shown in Fig.4 above, and the work hardening characteristics of all the Nb alloys being discussed in this paper are plotted in Fig.5 for comparison.

C. Subdivided Element

One of the difficulties encountered to achieve the HEP goal with the design of the conductor currently manufactured at OKAS is, to meet the effective filament diameter (d_{eff}). Restack wire has a single diffusion barrier and very high Nb content, hence it is very difficult to prevent the adjacent subelements from bridging together during the reaction [10].

Recent work done under the SBIR subcontract from Supergenics has verified that the subelement material co-extruded with the pure Nb barrier can be drawn down to 0.15mm without having much trouble [11]. Furthermore, the non-Cu J_c of 2700 A/mm² at 12T has been obtained in the fully coupled cable sample with seven of 0.15mm diameter strand.

The concept of this co-extruded subelement was employed in the billet design of the material for the production order from LBNL. This material, however, contains dual barrier of Nb and Ta. Nb in the inner layer is expected to become superconductor after reaction to produce extra current. Fig.6 exhibits the cross section of 51.6 mm diameter extruded rod. The composition of the elements has been re-adjusted accordingly to achieve the maximum J_c possibly obtainable within the design. It is anticipated that material from this billet will produce a reasonably reduced d_{eff} and the J_c of 2400 A/mm² at 12T in the non-Cu.

Fig. 6. A partial cross section of subelement rod co-extruded with Ta/Nb barrier.

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Work Hardening of Nb Alloys

Fig. 5. A comparison of the work hardening of Nb alloys used for the material described in this work.

DEVELOPMENT OF Nb₃Sn WIRES MADE BY THE INTERNAL-TIN PROCESS

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ABSTRACT

A survey is given of the test results obtained on the internal-tin Nb₃Sn strand produced by IGC for the Central Solenoid Model Coil (CSMC) program for the International Thermonuclear Experimental Reactor (ITER). Approximately four tonnes of strand have been fabricated, tested and the data analyzed. The results show a relatively small variation in the measured properties.

The standard ITER strand design has also been modified by changing the Cu and Sn percentage, the composition, size and spacing of the filaments, and the size and chemical nature of the barrier. As a result of this work, a range of materials, each with significantly different properties, was produced. One such material met the original HP 1 specification more completely than earlier material. Another met the HP-2 specification and a third was used successfully in one of the inner coils of D20, the world's highest field dipole, recently tested at Lawrence Berkeley National Laboratory (LBNL).

INTRODUCTION

Approximately four tonnes of Nb₃Sn material have been manufactured by the internal-tin process for use in the US section of the CSMC for ITER. The primary aim of this work was to produce on a commercial scale, a large volume of material with reliable properties. The extent to which these properties varied throughout the production cycle, could be determined relatively easily as every length supplied was rigorously tested. For these reasons the minimum number of changes was made in the process during the production phase, even though it was known from the start of Stage IV that the J_c may be below the HP 1 specification. A parallel program has, however, been carried out in order to develop material to more easily meet the original ITER HP 1 and HP 2 specifications, shown in Table 1.

The production work has now been successfully completed, and material has been made which meets these original specifications. Sufficient property margin now exists so that there is considerable confidence that large quantities can be made with all the properties meeting either the HP 1 or the HP 2 specifications.

Also reported here is an additional modification of the ITER type of material made to meet the requirements of the D20 high field dipole developed at Lawrence Berkeley Laboratory (LBNL).

Table 1. ITER strand specifications

Property	HP I	HP II
Strand diameter	0.81 mm	0.81 mm
Cr-plating thickness	2 μm	2 μm
Cu/non-Cu ratio	1.5	1.5
RRR at 0 T, $p(273\text{ K})/p(20\text{ K})$	> 100	> 100
Twist pitch (right hand)	$\leq 10\text{ mm}$	$\leq 10\text{ mm}$
Non-Cu J_c at 12 T, 4.2 K, 0.1 $\mu\text{V/cm}$	> 700 A/mm ²	> 550 A/mm ²
Non-Cu hysteresis losses for $\pm 3\text{ T}$ cycle at 4.2 K	< 600 mJ/cm ³	< 200 mJ/cm ³
n value at 12 T, 4.2 K, 0.1 $\mu\text{V/cm}$	> 20	> 20

TEST RESULTS ON STRAND FOR THE US SECTION OF THE CS MODEL COIL

The design of the IGC strand and some of the results of Stage III of the development program to produce 500 kg of strand were presented two years ago at CEC/ICMC in Columbus¹. In a subsequent paper², the J_c and loss properties of all the Stage III material were given, together with the preliminary results on Stage IV. Although the Stage III results segregated into four widely separated groups, tighter raw material specifications and standardized design and manufacturing details reduced property variations, as evident in the preliminary results of Stage IV.

The work reported here is on all the Stage IV material and it confirms the relatively small spread in the data. It shows, however, the necessity to control closely the dimensional parameters of the raw materials and all the aspects of the fabrication process.

165 restack billets were made to supply the needed 3.72 tonnes, and these were all tested on both ends, for J_c , n value, Cu/non-Cu ratio, and RRR. Since early results showed losses to be well below the HP 1 specification of 600 mJ/cm³, the US Home team required that only one in five of the restacks be tested for losses in the later work.

The manufacture of the production lots was carried out in 13 groups of approximately 13 restacks each. Two small changes were made in the strand design, and these were applied to groups 7-13. One was to increase the amount of Cu in the stabilizer and the other to increase the diameter specification on the superconductor rods from the mean value to the upper end of the original specification. This amounted to a 3.15 % increase in the Nb 7.5 wt.% Ta content. These small modifications changed the mean J_c at 12T from 685 A/mm² to 690 A/mm², i.e. less than expected. This could be due to the fact that no corresponding change was made in the Sn content when the rod diameter was increased. Insufficient Sn could also account for the fact that instead of losses increasing, the average decreased slightly from 445 mJ/cm³ to 430 mJ/cm³. These results again emphasize the critical nature of the Sn content.

Figures 1 & 2 show histograms of the J_c and loss data for the groups 1-6 and 7-13 indicating the small shifts in the mean values due to the changes discussed above.

The total number of strands shipped from the 165 restacks was 254 and approximately half of the material shipped was in lengths greater than 4 km. Two RRR values were measured on each of the restacks shipped. The mean value was 122. In only four of the groups were there readings below 100, Figure 3. Many different furnaces were used for the heat treatment of the samples and the RRR values obtained appeared to be related more to the quality of the furnace atmosphere than to that of the wire.

PROPERTY CHANGES IN ITER TYPE STRAND

From the results of the Stage III work, it was obvious that IGC would be unlikely to meet the 700 A/mm² at 12T specification, without some changes in the design of the strand. No such change was permitted at that stage, instead, a specification variance was made to

Figure 1. Distribution of J_c values for the two categories, group 1-6 and group 7-13.

Figure 2. Distribution of loss values for the two categories, group 1-6 and group 7-13.

Figure 3. RRR values for the various groups of Stage IV material.

Figure 4. Losses versus J_c for a variety of different strand designs.

allow the acceptance of material with a $J_c > 650$ A/mm². Although the material was originally designed to meet the HP 1 specification, presumably for TF coil applications, later in the program the plan was to use the material in the CS model coil where losses were of greater importance and the HP 2 specification perhaps a more appropriate target. Since losses increase with J_c , other factors being constant, by permitting a lower J_c the coil designers were assured that the losses would not increase further. The J_c and loss results for Stage IV are shown in Figure 4 enclosed by the ellipse. As expected, the average J_c lies slightly below 700 A/mm². Groups 1-6 are represented by the open square symbols and groups 7-13 by the filled circles.

It was pointed out from the results of the first round of strand benchmark tests^{3,4}, that the H_{c2} and T_c of the IGC ITER strand were lower than those of some of the materials from other manufacturers. These low properties at high fields were corrected by the addition of Ti to the Sn^{2, 5}, although such changes could not be made on the Stage IV strand. Not only does the Ti increase the high field J_c properties but, since it increases the grain size of the Nb₃Sn, the grain boundary pinning is reduced and the J_c lowered in the low field region. This, in turn, reduces losses as measured over the $\pm 3T$ cycle.

A further method of decreasing the losses was also described earlier². That was by replacing the Nb backed Ta barrier by a single Ta barrier. Thus Nb peaks in the low field region of the magnetization curve, which have a significant effect on the losses as measured in the $\pm 3T$ region, are eliminated. This replacement was introduced in two different ways, by:

1. replacing the Nb with Ta, and the other
2. reducing the thickness of the barrier, in addition to replacing the Nb with Ta, to about 2/3 that of the original ITER design. The second approach raised the J_c as it increased the amount of material contributing to the I_c . The effect of making this change alone, i.e. employing an all-Ta barrier of 2/3 the thickness of the original in the standard ITER design, is shown by the point noted as "Mod. ITER" in Figure 4. This is well within the original HP 1 area.

The J_c and losses obtained appear to be equal, at 12 T, to those of the intermediate density (ID) material (see below) with Ti added to the Sn. It may be important to note that, when the filament diameter was increased in ID, the Sn content was not changed proportionately.

In earlier work^{2, 6}, it has been pointed out that one of the most effective ways of reducing losses is to reduce the size of the Sn area in the center of the subelement. This reduces the bridging that occurs in the inner ring of filaments. Unfortunately, unless the Sn content is increased in other areas of the assemblage⁷, the J_c is lowered significantly.

INTERNAL TIN MATERIAL MEETING THE HP 2 SPECIFICATION

In a previous paper² we stated that we expected to be able to meet the HP 2 specification without any major change in the subelement design. This has now been achieved by modifying the material made for ITER in the following manner. The area of the filaments was increased by 10%, with only a 6% increase in Sn, (ID), this increased both J_c and losses. The Sn was then lowered by 17% (ID LSn) and this reduced both J_c and losses considerably. The Nb was removed from the usual Nb/Ta barrier used in the ITER design and replaced by Ta (ID TaLSn). The percentage of the core volume occupied by the barrier was less, in this particular sample, than that of the standard ITER design. This contributed to an increase in J_c and resulted in a material which had properties almost in the top right hand corner of the HP 2 box. Since no Ti was added to the Sn in this material, it is expected that its H_{c2} and T_c will be below that of similar material made by several other manufacturers. Finally, Ti was added to the Sn (ID TiTaLSn), giving a material with properties well within the HP 2 specification without changing the subelement design significantly from that of ITER. In this material, the stabilizer contained an all-Ta barrier, the thickness of which was equal to that of the dual barrier of ITER. While the loss advantages of the Ta were achieved, the J_c improvements noted in ID TaSn and Mod ITER were not observed.

A modified design, using a thinner all-Ta barrier, is presently being made in an effort to achieve a $J_c > 700 \text{ A/mm}^2$ and losses $< 200 \text{ mJ/cm}^3$.

MATERIAL FOR D20

The Lawrence Berkeley National Laboratory (LBNL) has recently completed and tested a 50mm bore, one meter long dipole which achieved a field of 13.5T at 1.8K. This is a world record for a high field dipole. One of the two inner windings was made from an IGC strand, a cross section of which is shown in Figure 5, and the other from Teledyne Wah Chang (TWC) material. The requirements for the strand differ from those of the HP 1 and HP 2 materials. A higher J_c was needed, as was a lower Cu/non-Cu ratio and losses were of less importance than for fusion applications. The Cu percentage was therefore reduced from 60 vol. % to 30 vol.% and the filament area increased 17% and while the Sn was increased it was not in proportion to the superconductor change. The material gave a J_c of 850 A/mm^2 at 12T and losses of 850 mJ/cm^3 . The barrier used was a Ta lined Nb barrier, similar to that used in the standard ITER strand.

A higher J_c could have been achieved by employing an all Nb barrier, but as reported earlier⁸, such a barrier can lead to low field instabilities. This is shown in the magnetization curve in Figure 6. Since this material was supplied to LBNL, IGC has developed a new barrier design, in which the continuous loop of Nb_3Sn formed with an all Nb barrier is interrupted. In this way the low field instabilities are eliminated without a serious loss in J_c . The magnetization curve for such material is shown in Figure 7. While the losses are much greater than those in the case of the Ta-lined Nb barrier or the all Ta barrier, (Figure 8), no low field instabilities are observed.

The successful operation of D20 indicated that the strain limitations of Nb_3Sn are not a serious factor, and that Rutherford cables of internal-tin can be made to operate successfully in dipole magnets.

SUMMARY

The properties of internal-tin Nb_3Sn for the ITER CS model coil have been described, and the variation of J_c and losses was $\pm 7\%$ and $\pm 13\%$, respectively, in approximately 4 tonnes of material manufactured. It is expected that this spread in the data will be reduced somewhat in future production lots. The work illustrates that quantity production of this type

Figure 5. Cross section of the IGC strand used in the inner coil of D20.

Figure 6. Magnetization curve for material containing a 100% Nb barrier. The relatively large flux jumps in the low field region are very apparent.

Figure 7. Magnetization curve for material containing an interrupted Nb barrier. No flux jumps in the low field region are observed.

Figure 8. Magnetization curves for 1. material containing a Nb backed Ta barrier, as in the standard ITER strand and 2. material with a 100% Ta barrier.

of material can indeed be accomplished and reliable and reproducible properties obtained. The flexibility of the internal tin process has been shown by the development, using a slightly modified standard ITER subelement design, of material meeting the HP 1 and HP 2 specifications. Also, the same basic design has been used successfully in one of the inner coils of D20, the LBNL 13.5 T high field dipole.

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PROPERTIES OF INTERNAL-TIN Nb_3Sn STRAND FOR THE INTERNATIONAL THERMONUCLEAR EXPERIMENTAL REACTOR*

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ABSTRACT

We report on the design and properties of a Nb_3Sn wire strand developed for the International Thermonuclear Experimental Reactor (ITER). The internal-tin process was employed using 19 subelements, 6 spacers, and a Ta-containing barrier to separate the superconducting core from the Cu stabilizer. Specific values of the four properties — critical current density J_c , hysteresis losses, residual resistivity ratio RRR, and piece length — required by the ITER specification are difficult to achieve simultaneously in one strand design. This is particularly true when the strand is Cr plated to prevent sintering and to provide interstrand resistance. Some aspects of conductor design and heat treatment, and how these affect the various properties, including n value, are outlined.

INTRODUCTION

The International Thermonuclear Experimental Reactor (ITER) Joint Central Team (JCT), consisting of representatives from Europe, Japan, the Russian Federation, and the United States, has launched a program to manufacture two model coils. These consist of (1) a central solenoid (CS) model coil with inserts requiring over 25 metric tons (t) of Nb_3Sn strand to be incorporated in 54 t of a thick-walled Incoloy 908 square jacket, and (2) a toroidal field (TF) model coil requiring 3.6 t of Nb_3Sn strand and 1 t of circular thin-walled Incoloy 908 tube.

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Table 1. ITER strand specifications, after Bruzzone et al.¹

Property	HP I	HP II
Strand diameter	0.81 mm	0.81 mm
Cr-plating thickness	2 μm	2 μm
Cu/non-Cu ratio	1.5	1.5
RRR at 0 T, $\rho(273\text{ K})/\rho(20\text{ K})$	> 100	> 100
Twist pitch (right hand)	$\leq 10\text{ mm}$	$\leq 10\text{ mm}$
Non-Cu J_c at 12 T, 4.2 K, 0.1 $\mu\text{V/cm}$	> 700 A/mm ²	> 550 A/mm ²
Non-Cu hysteresis losses for $\pm 3\text{ T}$ cycle at 4.2 K	< 600 mJ/cm ³	< 200 mJ/cm ³
n value at 12 T, 4.2 K, 0.1 $\mu\text{V/cm}$	> 20	> 20

Two types of Nb₃Sn strand have been specified, "High Performance I and II" (HP I and HP II), and the properties required of each of these are outlined in Table 1. They are effectively the same except for the critical current, J_c , and loss requirements.¹

IGC, at the request of the U.S. Home Team, developed a strand to meet the HP I specification for initial use in the outer sections of the CS model coil. Stage III of this development has been completed and 500 kg of strand made. This paper describes some of the development work and results obtained.

STRAND DESIGN

The higher J_c requirements of the HP I specification are more easily met by the internal-tin process than by the bronze approach because of the higher concentration of Sn around the filaments. This advantage is offset by the need for closer control of the strand design, fabrication, and heat treatment because of their effect on the properties listed in Table 1.

When Nb₃Sn conductors are used in small DC magnets, factors such as piece length, hysteresis loss, RRR of the stabilizing Cu, and the effects of Cr plating are usually not even considered. Critical current density is the property most frequently emphasized, although in magnets for magnetic resonance imaging applications, n values are also specified. In the case of the ITER HP I specification, all requirements have to be met simultaneously, including a high J_c .

One property which needs particular attention in the IGC internal-tin process is piece length. Once Sn is introduced into the conductor assembly, all subsequent processing, prior to the final heat treatment, has to be performed cold. This means that the condition of the components, particularly the hardness and the state of the surface, have to be carefully controlled together with the processing parameters to ensure good bonding and thus acceptable piece length.

Heat treatment is also more critical in the internal-tin process. While it is taking place, not only are the superconducting properties being developed, but also Sn and Cu are interdiffusing. This leads to the need for the heating and cooling rates to be controlled throughout the cycle, in addition to the ultimate time and temperature of heat treatment.² The importance of this, when heat-treating large magnets, must not be overlooked because of the considerable mass of the structures involved. Although the ultimate aim is to have a common heat treatment for all ITER conductors, this has not yet been achieved, nor have the heat treatment ramp rates been specified.

The results of the early development work on the IGC ITER strand, carried out in Stage II of the program, are reported in Ref. 3. Five different designs were explored in the

Fig. 1. Schematic illustration of the strand used in Stage III of the ITER strand development program.

early work, and the effort was then concentrated on one of these, modified with three different spacers. We concluded that a design with a spacer containing a Sn core and no additional filaments gave a good piece length and met all the ITER HP I specifications. This was the design chosen for Stage III. It is shown schematically in Fig. 1, and a cross section before reaction is shown in Fig. 2. It is made up of 19 subelements, each containing 162 filaments of Nb 7.5% Ta by mass, each 4.2 μm in diameter, and containing six Sn-cored spacers. The strand also contains a Ta-lined Nb barrier in the stabilizer which, in turn, provides about 60% Cu by volume to the overall strand.

Fig. 2. Cross section of ITER strand at 0.81 mm diameter, before reaction

The principal design characteristic that distinguishes the IGC strand from other internal-tin ITER strands is the single non-Cu area separated from the concentric Cu stabilizer by one Ta-lined Nb barrier. Other designs have multiple non-Cu areas, each separated by a barrier from the interconnected Cu area.

This single non-Cu area is intended to conserve space, reduce the chance of barrier breakage, and reduce the tendency for interfilament contact or "bridging." This contributes to meeting the J_c and hysteresis loss requirements while maintaining a good RRR after Cr plating.

Stage III of the program required 500 kg of strand, which was made from four different subelement billets and 23 restack assemblies. A restack assembly consisted of 19 subelement rods, six spacers, and a stabilizer tube.

TEST RESULTS ON THE STANDARD STRAND DESIGN

The choice of this strand design was based on a relatively small amount of test data on a conductor of the same design as that is referred to here as the ITER standard, shown in Table 2 with a superscript 3. These are the same data reported as design "c" in Table I of Ref. 3. These J_c data were later found to be inaccurate because of an incorrect field calibration of the test magnet. This led to a series of calibration tests for critical current I_c involving NIST and several test laboratories, using a new holder designed by MIT and detailed instructions from NIST on how to perform the tests. The corrected data are shown in Table 2. This cooperative effort eliminated what had, up to that time, caused a considerable variation in the I_c results obtained at different laboratories. IGC, using the test facilities of the National Magnet Laboratory at MIT, emerged from this round of tests as one of the laboratories whose results agreed with those of NIST. IGC also instituted an extensive investigation of the measurement of Cu/non-Cu ratios. This led to a more reliable conversion from I_c to J_c . The J_c test data presented in this paper on conductor of the standard design were obtained by IGC at MIT; the hysteresis losses were measured at NIST.

Although the data given in the Soft 18 meeting³ were presented as well within the HP I specification, they were actually below the minimum J_c specification when correctly tested. The heat treatment cycle used at this time was 15°C/h to 375°C, hold at 375°C for 24 h followed by a ramp at 75°C/h to 660°C and hold for 240 h. Based on the work described in Ref. 2 the ramp rate was changed to 6°C/h to 660°C and hold for 240 h and this brought the material back into the HP I J_c specification.

As mentioned above, four different subelements were used in the Stage III work. Each of these contained Nb 7.5 % Ta by mass rods, 8.13 mm in diameter, from three different heats. Subelements 0 and 1 were made from rods from the same heat but different batches. The main differences in the heats were in the grain sizes and the hardness of the starting rods. The rods in subelement 4 were harder and had a finer grain size. They also showed a wider variation in both grain size and hardness but, with one or two minor exceptions, both these properties were within product specifications.

The subelement billets were all prepared in the same manner and processed under identical conditions. Heat treatment was carried out in the same furnace and under the same conditions: a ramp rate of 6°C/h, 240 h at 660°C, and a cooling rate of 25°C/h. In Fig. 3, the 12-T J_c and loss properties obtained were grouped according to the subelement used.

Material from subelement billets 0 and 1 showed slightly higher losses than material from the other two subelements, but all the 12-T J_c values of the restacks made from these

Table 2. Test data from earlier work³

Sample Designation	J_c (A/mm ²) at 12 T	J_c (A/mm ²) at 12 T (NIST)	n Value	n Value (NIST)	Loss (mJ/cm ³)
15T	745 ³	655	26 ³	20	479
22	755 ³	683	35 ³	22	433

Fig. 3. Relationship between the J_c and the losses of the strand used in Stage III of the ITER development program.

Fig. 4. Relationship between the J_c and n value for the strand used in Stage III of the ITER development program.

subelements were above the specification and varied $\pm 5\%$ about 740 A/mm^2 whereas the losses varied only $\pm 3\%$ about 440 mJ/cm^3 . Material from subelement billet 4. showed slightly higher J_c and significantly lower losses, but the spread in values is approximately the same as in the material made from the other subelements. Material from subelement billet 3 showed intermediate losses, but J_c values were below the specification.

The n values plotted against J_c are shown in Fig. 4. Although there is scatter in the data, all the results are above the specified value of 20. The highest values were obtained in material made from subelements 0 and 1. The lowest values are the ones associated with the material from subelement 3. The subelement 4 values were intermediate between the other two groups.

Only a limited number of wire breaks occurred during the manufacture of material made from restacks employing subelements 0, 1 and 3. Several unbroken lengths of $>5000\text{m}$ were produced. The restacks from subelement 4 experienced greater wire breakage. In an effort to reduce this, CuSn spacers were replaced by pure Cu spacers in restacks 17-1 and 17-3. Breakage was not reduced significantly however, but the J_c was lowered somewhat.

After Cr plating and heat treatment for 240 h at 660°C with a ramp rate of 6°C/h and a cooling rate of 25°C/h , each restack must be tested for RRR. So far, only representative samples have been tested from restacks made with subelements 0, 1 and 3. These are restacks 03, 05 and 12 and the RRR values obtained at Brookhaven National Laboratory were 115, 135 and 129, respectively. All are above the minimum of the ITER specification.

"HIGH DENSITY" MATERIAL

It is obvious from the above that the internal-tin process is quite sensitive to the condition of the material in the subelement or to, as yet unknown, minor changes in the subelement design or manufacture. If our aim is to reliably and economically produce large quantities of material to the HP 1 specification, we must determine the cause of this variability. We are now carrying out investigations with this aim in mind,² but meanwhile, a practical, if less elegant, solution is to increase the J_c properties of all the material. In this way, even material from the worst subelements would still meet all the ITER HP 1 specifications. While such J_c increases will also increase the losses, Fig. 3 shows that these losses can be increased in all the subelements substantially before the HP 1 specification limit of 600 mJ/cm^3 is exceeded.

One way of improving the current carrying capacity of the existing material is to increase the time at 660°C from 240 h to 264 h. This has been shown, in the past, to increase the J_c about 5%. It does, however, allow increased Cr diffusion to take place and the RRR begins to approach the minimum of 100 in the Cr-plated strand.

A second way of improving the overall J_c is to redesign the subelement billet so as to increase the amount of superconductor in the non-Cu area. At the time that we became aware that a low J_c may be obtained from material made from subelement 3, we had available at IGC "high density" subelement material made for another application. We decided to make a 60 vol.% Cu restack to determine how closely the HP 1 requirements could be approached using such material. The term "high density" refers to an increase in the amount of Nb 7.5 wt.% Ta alloy in the non-Cu portion of the conductor. The high density material has more superconductor than does the standard ITER strand reported above. This density increase was accomplished by simply enlarging the rod diameter by 8% and increasing the size of the Sn pool slightly. This diameter change, of course,

decreases the spacing between the filaments and increases the extent to which filament contact occurs. Decreased spacing, however, may have a very important additional effect: The closely spaced filaments support one another, thus reducing "sausaging" which in turn raises J_c and the n values in a manner similar to that reported almost a decade ago for NbTi.^{4,5}

After a heat treatment of 240 h at 660°C with a ramp rate of 6°C/h, the high density material had a J_c of 893 A/mm² in the non-Cu and a loss of 690 mJ/cm³. It is obvious that the amount of superconductor was increased more than necessary and the ideal solution would be to use an intermediate-sized rod. We are carrying out such experiments at the present time.

Another possible approach exists, however, to bring the properties of the high density material into the HP 1 specification: change the heat treatment. When one has a material with a higher J_c than is required, it is possible to lower both the J_c and the losses to some extent by simply changing the heat treatment conditions. Doing this lowers the extent to which chromium diffusion takes place and this enables a high RRR to be maintained. It is much easier to move from the top right hand corner to the center of Fig. 5 without a design change than from the bottom left to the center when limitations exist on how aggressive the heat treatment can be.

Originally, the ITER JCT proposed that all Nb₃Sn strands for the model coils should have a common heat treatment, so that materials from different vendors could be mixed in the various coils. The heat treatment suggested was 650°C for 175 h. IGC found that, with the standard material, this heat treatment did not give the desired J_c and we therefore employed the one described above. A limitation on the extent to which the time and temperature can be further increased is imposed by the necessity to maintain the value of the RRR above 100 for heat-treated and Cr-plated strand.

The high density material gives us the opportunity to lower the time and temperature and vary the ramp rate. This was done in a series of steps and the results are shown in Fig. 5. Reducing the time at 660°C from 240 h to 175 h while maintaining the slow ramp rate appeared to have a similar effect to lowering the temperature from 660°C to 650°C while maintaining the time at 240 h, that is, little affect on the losses but a reduction in J_c . Lowering the time at 650°C from 240 h to 175 h appeared to change the J_c less than the losses. Increasing the ramp rate to 75°C/h lowers both losses and J_c and almost brought the "high density" material into the HP 1 specification with the JCT recommended heat treatment. Decreasing the time at 650°C to 79 h produced a material meeting all the HP 1 specifications within a reasonable margin. When the material was heat treated for 20 h at 700°C, it gave even better J_c with lower loss values, but the RRR on Cr-plated and heat-treated samples has not yet been determined.

Whether or not a ramp rate of 75°C/h is practical for the full sized ITER coils is uncertain, but the ramp rate is an important variable that must be specified and controlled if the data on the strand are to be meaningful for the magnet performance. All the "high density" data presented earlier in this paper, were obtained from one restack of one subelement billet. Most of the I_c measurements and all the loss measurements on this high density material were carried out at NIST.

EFFECT OF VARIOUS PARAMETERS ON n VALUE

In addition to the higher J_c values, the high density material has another improved property over the standard material. This is shown in Fig. 6, where n values for the high

Fig. 5. Relationship between the J_c and the losses of "high density" strand after various heat treatments compared with that of standard material for Stage III of the ITER development program.

density material after various heat treatments are plotted against 12-T J_c . As the aggressiveness of the heat treatment decreases, so does J_c and n but both are higher than the average of the data for the standard material when the heat treatment is the same, 660°C for 240 h and a ramp rate of 6°C/h. The data for the standard material were obtained by averaging the results from the same samples as those shown in Fig. 4. This increased n value is to be expected as the filaments are larger, but possibly the fact that the spacing between these filaments is also less may be of some significance. This has been shown earlier to be a very important factor controlling the "sausaging" of filaments in

Fig. 6. Relationship between n value and J_c for "high density" strand after various heat treatments compared with that of standard material after 660°C for 240 h.

Fig. 7. Relationship between n value and field for "high density" strand after various heat treatments compared with that of standard material after 660°C for 240 h.

multifilamentary NbTi wire.^{4,5} In Nb₃Sn, where the spacing has to be much greater than in NbTi in order to reduce filament contact, the effect may not be as great, but obviously it deserves further investigation. In Nb₃Sn, as with NbTi, n is often taken as a measure of filament quality, particularly by those concerned with persistence in NMR-type magnets.

Some of these users also specify that partial reaction be used in order to keep a ductile Nb core in the filaments to improve mechanical properties and assist in the making of persistent joints. The data shown in Fig. 6 demonstrate that these two requirements may lead to erroneous conclusions as they indicate that, as soon as the heat treatment falls below that for complete reaction, the n value falls rapidly.

A wide range of n values can be obtained on identical wire made from the same subelement and restack by varying the heat treatment. The n value is determined by both the longitudinal uniformity of the starting Nb 7.5 % Ta by mass filaments and the longitudinal uniformity of the reacted part of the filaments. These data indicate that partially reacted filaments must have some nonuniformity along the length. As the time and temperature are increased, a more uniform degree of reaction can be assumed and the n values improve. Similar effects of heat treatment on n value exist at other fields as shown in Fig. 7.

CONCLUSIONS

IGC has successfully completed Stage III of the ITER strand development program by producing 500 kg of strand meeting the HP I specification and is now starting on Stage IV, the production of the material for the model coil.

In Stage III, the criticality of many of the design, heat treatment, and manufacturing variables, particularly for the subelements, have been determined. IGC is working with the University of Wisconsin and an alloy manufacturer, to establish reliable quality assurance specifications for both raw materials and the various processing parameters to ensure less variability in the strand properties.

The importance of specifying the ramp rate to be used in the heat treatment of the test samples, to simulate that which can be achieved with both the model coils and the full-size coils, is obvious from the results reported. It is also important to specify the maximum and minimum times at temperature since too short a time will not be practical for large coils and too long a time may result in RRR degradation and possibly cause other problems. These latter constraints apply irrespective of the Nb₃Sn manufacturing process used.

N values can be influenced significantly by not only strand design but also heat treatment procedures and they should not be considered simply as measures of filament "sausaging." The *n* value is determined by both the longitudinal uniformity of the starting filaments and the longitudinal uniformity of the reacted part of the filaments.

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Some Effects of Matrix Additions to Internal Tin Processed Multifilamentary Nb₃Sn Superconductors

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Abstract — Internal tin processed Nb₃Sn multifilamentary wires were fabricated with matrices containing either Ti or Ge. Some effects of these matrix additions on the formation rate of Nb₃Sn and on the J_c's obtained, have been determined. The effect of a 0.7 wt.% Ge addition on material with Nb 1 wt.% Ta, Nb 7.5 wt.% Ta and pure Nb filaments has been examined as has that of three different levels of Ti in the Cu matrix with the same filament compositions. The tubular-tin-source (TTS) process was chosen for this work, because this process was believed to be less sensitive to the hardness of the matrix than the tin-core process. In this study, three different reaction heat treatments were carried out to explore the variation of J_c of these materials. A discussion of the results and significant effects of the addition elements is given.

I. INTRODUCTION

In Nb₃Sn multifilamentary superconductors, particularly when made by the internal-tin process, the properties and the ease with which the material can be fabricated into long piece lengths are related to the hardness of the core material when they are finally assembled. Various levels of titanium and germanium have been added to the copper matrix which surrounds the filaments and the effect of these on ductility and J_c are discussed and compared with those of the conventional pure copper matrix. Since our concern is to increase the J_c in the higher field region, the effect on material with three different compositions of filaments was also examined in this work.

Materials have been fabricated with Cu 0.7 wt.% Ge, Cu 0.4 wt.% Ti and Cu 0.9 wt.% Ti as well as pure Cu matrices and examined metallurgically and electrically after a series of different heat treatments. The Ge appears to slow down the formation of Nb₃Sn but, nevertheless, it increases J_c significantly, presumably due to the fact that it promotes a smaller grain size.

The rate of strain hardening of matrix materials containing Ge and Ti is expected to be higher than that of pure copper and, in the past, problems have been encountered when attempts were made to cold bond hard subelements in the tin-core process. The TTS process [1] has, therefore, been chosen for its reduced sensitivity to the hardness of the matrix as a hot-isostatic-pressing step is used in the processing of the core, and only a few surfaces require cold bonding.

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II. BACKGROUND

It has been reported [2], [3] that, when a 7 at.% Sn bronze containing 0.5 at.% Ge is used to replace the normal 7 at.% Sn bronze as the matrix of single-core composite tapes in the bronze process, J_c of the Nb₃Sn layer increased by a factor of ten in the field range 8 to 16 T compared with that of Nb/Cu 7 at.% Sn. By the simultaneous addition of 0.5 at.% Ge to the matrix and 1 at.% Ta or Hf to the core, the J_c of Nb₃Sn layer approached 10⁶ A/cm² at 8 T, even where the Nb₃Sn layer is relatively thick. An SEM photomicrograph of the fractured surface of the cross section of Nb 0.5 at.% Ta/Cu 7 at.% Sn 0.5 at.% Ge composite heat treated at 775°C for 100 hrs. showed a much smaller grain size than those in the matrix without Ge presence.

The early work at IGC/AS was done using the 19 subelement material with Nb 1.3 wt.% Ti filaments [4]. In this work J_c properties of the material were good but the piece length was poor. The rapid work hardening of these Ti containing filaments was, at the time, believed to be one of the primary factors contributing to the piece length problem. It was felt that, by introducing the Ti from the matrix as opposed to incorporating it in the filaments initially, the piece length problem could be overcome. Before this could be proved it was decided to determine the feasibility of producing high J_c internal-tin material when the Ti was introduced to the matrix. Also the Ti alloy is believed to be more responsive to different heat treatments since the Ti tends to speed up the diffusion rate of Sn in Nb. For these reasons there is considerable incentive to examine methods of improving both the electrical and mechanical properties in the Ti doped materials.

III. EXPERIMENTAL PROCEDURE

A. Material Preparation

This work compares, on a trial billet scale, the properties of Nb₃Sn products made from Cu 0.7 wt.% Ge, Cu 0.4 wt.% and 0.9 wt.% Ti matrices with those of a pure Cu matrix. Simultaneously pure Nb, Nb 1.0 wt.% and 7.5 wt.% Ta filamentary materials were investigated.

Cast alloys containing Ge and Ti were cross grain forged to a 54.6 mm diameter rod. This was gundrilled with 60 holes in a 4 row circular pattern [5]. A similar billet was prepared from OFHC copper to provide comparative data. Sixty rods of Nb, and an equal number of Nb 1.0 wt.% Ta and Nb 7.5 wt.% Ta, as annealed condition, each 4.06 mm in diameter were inserted into the holes. The three types of rods together with the four matrix compositions required the production of twelve billets, each containing a different filament-matrix

TABLE I
COMPOSITIONS OF MATERIALS STUDIED IN THIS WORK

Matrix	Filament	Combination
Pure Cu	Pure Nb	Nb/Cu
	Nb 1.0 wt.% Ta	1.0 Ta/Cu
	Nb 7.5 wt.% Ta	7.5 Ta/Cu
Cu 0.7 wt.% Ge	Pure Nb	Nb/0.7 Ge
	Nb 1.0 wt.% Ta	1.0 Ta/0.7 Ge
	Nb 7.5 wt.% Ta	7.5 Ta/0.7 Ge
Cu 0.4 wt.% Ti	Pure Nb	Nb/0.4 Ti
	Nb 1.0 wt.% Ta	1.0 Ta/0.4 Ti
	Nb 7.5 wt.% Ta	7.5 Ta/0.4 Ti
Cu 0.9 wt.% Ti	Pure Nb	Nb/0.9 Ti
	Nb 1.0 wt.% Ta	1.0 Ta/0.9 Ti
	Nb 7.5 wt.% Ta	7.5 Ta/0.9 Ti

combination. Compositions of the materials investigated in this work are listed in Table I.

All twelve billets were electron-beam welded and hot isostatically pressed, then extruded at 700°C to 12.7 mm diameter rods. These rods were then wrapped with tin sheet followed by Cu foil. These wrapped rods were then inserted into stabilizer tubes, with a Nb-Ta barrier as the inner surface. The assemblies were drawn down to 0.41 mm diameter wires with no drawing problems or wire breakage. The average filament size in the finished wires was 17 μ m. The copper stabilizer occupied 55% of the wire cross section. A cross section of one of the finished wires prior to a reaction heat treatment is shown in Fig. 1.

The wires were heat treated in two different reaction cycles in the following manner. The first condition included 200°C for 96 hrs, 375°C for 24 hrs, 580°C for 48 hrs and 700°C for 48 hrs. This heat treatment is believed to convert the material under the barrier into a relatively homogeneous high-tin bronze. The second reaction condition included 375°C for 24 hrs followed by 650°C for 240 hrs. While the material with a Cu matrix showed filaments which were almost reacted throughout, particularly with the second heat treatment, the Cu 0.7 wt.% Ge matrix showed filaments reacted only about 65%. This shows that the rate of formation of Nb₃Sn is much slower in the presence of Ge in the matrix and this was particularly noticeable in the material also containing 7.5 wt.% Ta in the filaments. This is presumably due to the build-up of Ge at the interface between the filaments and the matrix [3].

Fig. 1. Overall cross section of a TTS wire of Cu matrix with Nb filaments.

B. Work Hardening Characteristics

While previous investigators [3] have reported that the workability of bronze was not significantly affected by the presence of a small amount of Ge, there has been no report of the affect of this element on the workability of Cu. For this reason we undertook an examination of the work-hardening characteristics of the matrix materials prepared in this investigation. Fig. 2 shows graphically the Vickers Hardness Numbers (VHN) taken after various strains. These show that the effect of 0.7 wt.% Ge and 0.4 wt.% Ti on the work hardening of Cu is relatively small and this predicts that no significant problems will be experienced in the fabrication of the wire with matrices containing these alloys. On the other hand, the material with 0.9 wt.% Ti work-hardens in the early stage of straining at a far greater rate than the others although no significant drawing related problems were encountered on this material.

IV. EXPERIMENTAL RESULTS

From the observation of the wire cross section after a series of heat treatments, there are wide differences in the amounts of reaction that take place in the relatively large diameter filaments of the material with different compositions. For this reason a simple determination of J_c in the non-Cu region does not always yield complete information on the effect of compositional changes. Table II shows these non-Cu J_c properties for all the different compositions after the two heat treatments together with the percentage of the filaments that are reacted. The materials heat treated at 650°C for 240 hrs could be compared on the basis of non-Cu J_c , after extrapolating those values to 100% reaction, since the materials made with Ge in the matrix show relatively low reacted area in the filaments whereas the filaments in all other cases are almost completely reacted. These non-Cu J_c 's of selected compositions at various fields for the materials heat treated at 650°C for 240 hrs are shown in Fig. 3.

Fig. 2. A comparison of the work hardening characteristics of the matrices.

TABLE II

NON-Cu J_c AND PERCENTAGE OF FILAMENT REACTED AFTER DIFFERENT HEAT TREATMENT

Composition	Non-Cu J_c (A/mm ²)							
	650°C / 240 hrs				700°C / 48 hrs			
	8 T	10 T	12 T	%	8 T	10 T	12 T	%
Nb/Cu	1992	1258	753	97	1533	1113	639	85
1.0Ta/Cu	1753	1108	665	87	1558	1036	660	81
7.5Ta/Cu	1889	1298	886	74	1327	942	685	65
Nb/0.7Ge	2423	1709	1173	70	1182	808	582	49
1.0Ta/0.7Ge	1917	1346	898	84	1139	774	514	56
7.5Ta/0.7Ge	2172	1575	1221	44	925	702	511	39
Nb/0.4Ti	2087	1650	1307	94	1707	1348	1063	90
1.0Ta/0.4Ti	1800	1275	961	92	1877	1366	953	91
7.5Ta/0.4Ti	1920	1422	1102	93	1848	1355	998	87
Nb/0.9Ti	1780	1358	1027	90	1721	1334	1028	94
1.0Ta/0.9Ti	2242	1698	1261	98	1854	1386	1065	93
7.5Ta/0.9Ti	1613	1230	922	97	1224	905	670	89

Fig. 3. J_c 's of non-Cu area plotted against the magnetic field for the samples heat treated at 650°C for 240 hrs.

It is clear from an examination of Table II and Fig. 3 that germanium containing materials show the highest non-Cu J_c 's at all fields between 8 and 12 T when compared with the corresponding Cu matrix materials. This is true whether Ta is present in the Nb or not. The titanium containing materials, particularly 0.4 wt.% Ti in the matrix, reveal themselves as the second highest J_c group. The best properties at 8 T are shown by the material with Nb filaments and a matrix containing Ge. At 12 T, the same Ge matrix with Nb 7.5 wt.% Ta filaments and 0.4 wt.% Ti matrix with pure Nb filaments shows the best results with insignificant difference. These are 27 % and 32 % higher than those of material with a

pure Cu matrix and Nb 7.5 wt.% Ta filaments, respectively.

An examination of the curves in Fig. 3 shows that the improvement in J_c from Ge additions to the matrix without 7.5 wt.% Ta is greater than when 7.5 wt.% Ta is present. If Ge is present, the 7.5 wt.% Ta shows superior J_c 's to the material with pure Nb filaments at fields above 11 T. With pure Cu matrix, the equivalent cross over is at 9 T. This fact could be significant in the choice of a conductor for TPX, where the specifications for the strand are at 8 and 9 T. From overall analysis of current densities against the heat treatments, it is assumed that the pure Cu matrix material has not been degraded by prolonged heat treatment.

A further examination of Table II and Fig. 3 shows that 700°C/48hrs heat treatment leads to lower non-Cu J_c 's than those shown by the 650°C/240hrs heat treated material. This is primarily due to the fact that the filaments are less completely reacted in the 700°C/48hrs heat treatment. This is particularly true in the case of the Ge containing materials and is most noticeable in the material with the alloyed matrix and 7.5 wt.% Ta in the Nb filaments. In order to determine more clearly what the Ge addition to the matrix does, the J_c 's in the Nb₃Sn layer have been measured. A correction to the values was made by assuming a 37 vol.% increase when the Nb₃Sn is formed [6]. The results are shown in bar graph form in Fig. 4, made at 12 T for the materials heat treated in both conditions.

Reference [7] defines the effective transition temperature (T_c^*) in the temperature correction equation. T_c^* at 12 T for these materials after the 650°C/240hrs heat treatment showed clearly higher (11.4K) values for the 7.5 wt.% Ta materials. In the absence of Ta and with a 1.0 wt.% Ta, the Ge containing materials showed slightly higher T_c^* values (10.4K) than the corresponding pure Cu matrix materials (9.8K). Contrary to the results of Tachikawa's work with bronze process, the effect of a small amount of Ta in the filaments and Ge in the matrix does not appear to be very remarkable. This effect may be less in our work than in theirs as they used 2 wt.% Ta as opposed to our 1 wt.% Ta.

For the materials with Cu or Cu 0.7 wt.% Ge matrix, it is more likely true that the heat treatment with the short time at 700°C after a hold at 580°C generally appears to give the higher J_c 's in the Nb₃Sn layer. The Ge and Ti containing materials show also higher J_c in the Nb₃Sn than the corresponding materials with pure Cu matrices. It is possible that, in materials with finer filaments, we may be able to take advantage of shorter heat treatment times or lower heat treatment temperatures which result in apparently higher J_c in the Nb₃Sn.

The reason for the higher J_c 's in the Ge containing materials has been previously attributed to a finer grain size [3]. In this investigation the grain size on the fractured surface of the wires after the 700°C/48hrs heat treatment was examined in the SEM for the materials with Nb 7.5 wt.% Ta filaments in the Cu and Cu 0.7 wt.% Ge matrices respectively. While the grain size of Nb₃Sn with Ge containing matrix is somewhat smaller than that with Cu matrix, the difference is not outstanding.

Fig. 4. J_c of Nb_3Sn at 12 T for various compositions and heat treatments.

V. CONCLUSIONS

The addition of Ge to the matrix appears to slow down the diffusion rate but the J_c in the reacted layer is higher than in material made from a pure Cu matrix. This is presumably due to the formation of a Ge-rich layer at the filament/matrix interface and it improves the J_c in the non-Cu area over the field range between 8 T and 12 T. Ti addition, on the other hand, tends to speed up the formation of Nb_3Sn at a relatively slow rate. The extent of the increase in J_c for the Ge containing material is not as great as is indicated in the previous work on bronze [3] but is, nevertheless, quite significant and it can probably be increased further by simultaneously optimizing composition, heat treatment and filament size. The cause for this J_c increase for the materials with Ge in the matrix is probably due to the corresponding decrease in grain size.

From the comparison of the results reported by Tachikawa [3], and those obtained in this investigation, it is obvious that the results in this study are not as spectacular as those reported for bronze. This could be due to the very high temperature and length of time used in their work. Another reason could be that the effect is more noticeable in material with a bronze matrix where the tin concentration is lower.

Since work hardening has been found to be small at the Ge and low Ti (1.0 wt.%) levels explored here, it is likely that this technique can be applied to larger strands made by the tin-core technique used in ITER [8] and TPX. In this process, low work hardening is required to ensure bonding of the subelements and thus will obtain reliable piece lengths.

Only one Ge level has been examined. This is not necessarily the optimum and the effect on the losses has not been explored here, although this has been done for the bronze matrix [9]. It is felt that in the case of internal-tin-processed Nb_3Sn , bridging between filaments is the main factor affecting losses and that additions to the matrix will have little effect on this. This assumption will be checked when finer filament material is made using the tin-core process.

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