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.

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, ρ(273 K)/ρ(20 K)	> 100	> 100
Twist pitch (right hand)	≤ 10 mm	≤ 10 mm
Non-Cu J_c at 12 T, 4.2 K, 0.1 μV/cm	> 700 A/mm^2	> 550 A/mm^2
Non-Cu hysteresis losses for ± 3 T cycle at 4.2 K	< 600 mJ/cm^3	< 200 mJ/cm^3
n value at 12 T, 4.2 K, 0.1 μV/cm	> 20	> 20

Two types of Nb_3Sn 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 Nb_3Sn 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

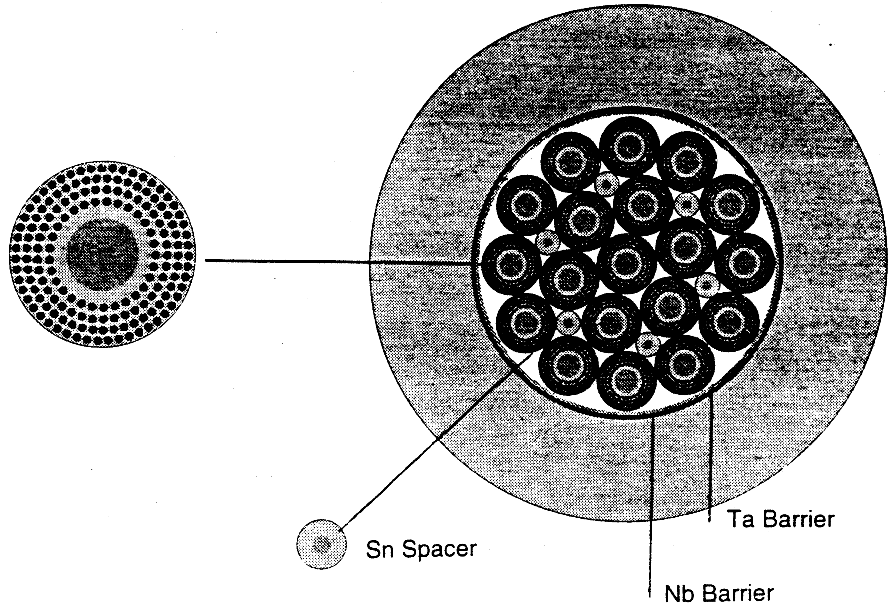

Fig. 1. Schematic illustration of the strand used in Stage III of the ITER strand development program.

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.

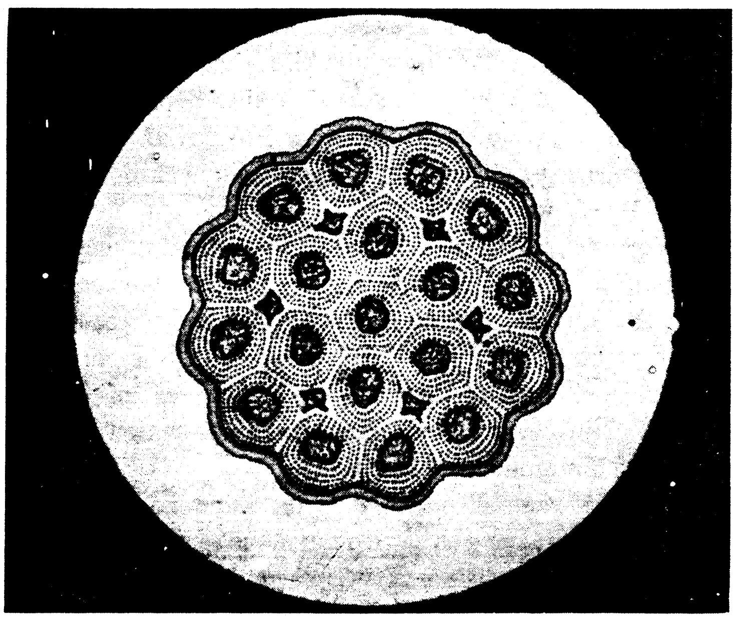

Fig. 2. Cross section of ITER strand at 0.81 mm diameter, before reaction

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 meeting[3] 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 work [3]

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

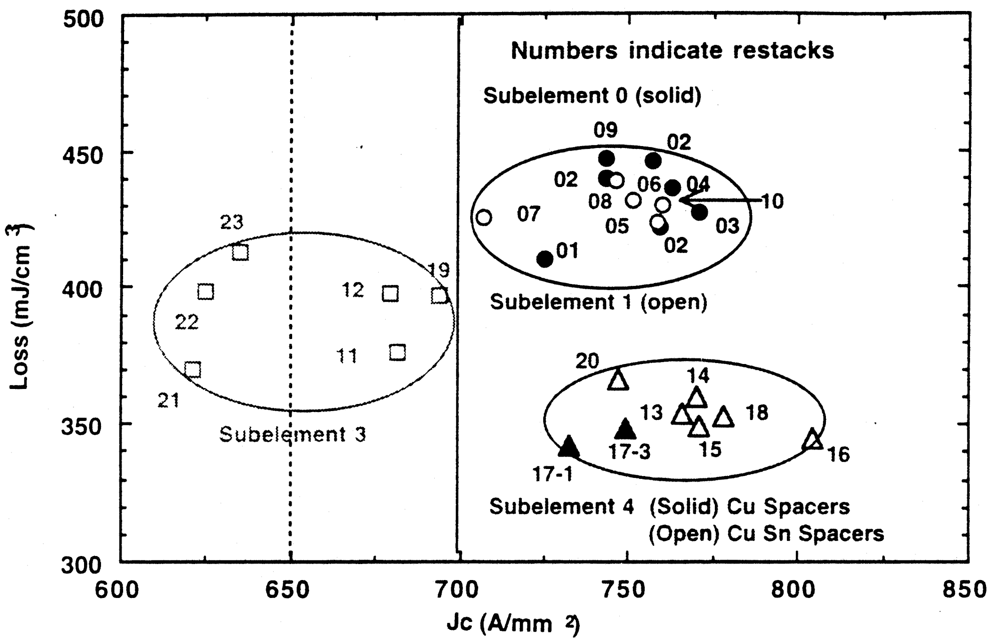

Fig. 3. Relationship between the J_c and the losses of the strand used in Stage III of the ITER development program.

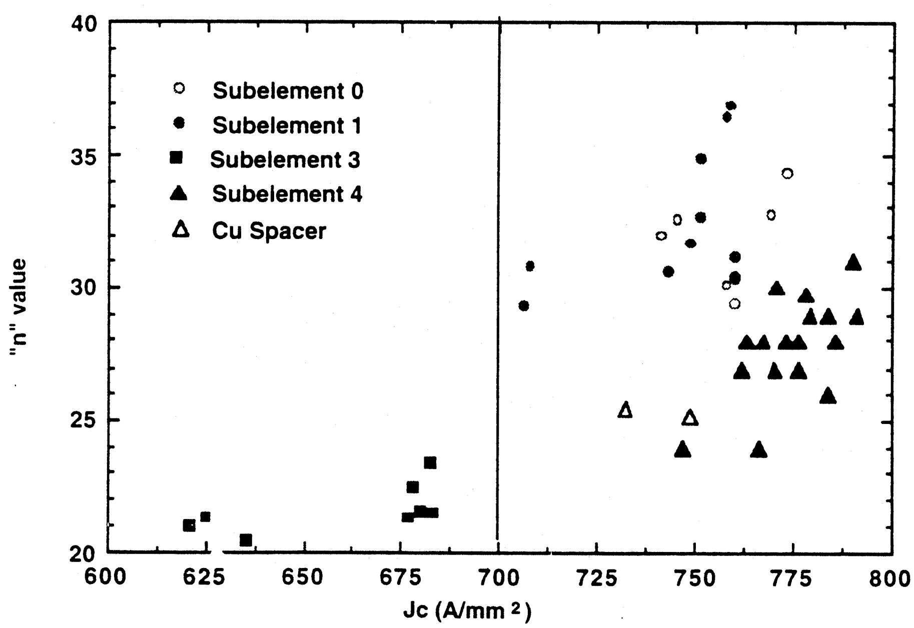

Fig. 4. Relationship between the J_c and n value for the strand used in Stage IIIof the ITER development program.

subelements were above the specification and varied ±5% about 740 A/mm^2 whereas thelosses varied only ±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 >5000m 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/mm^2 in the non-Cu and a loss of 690 mJ/cm^3. 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 Nb_3Sn 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

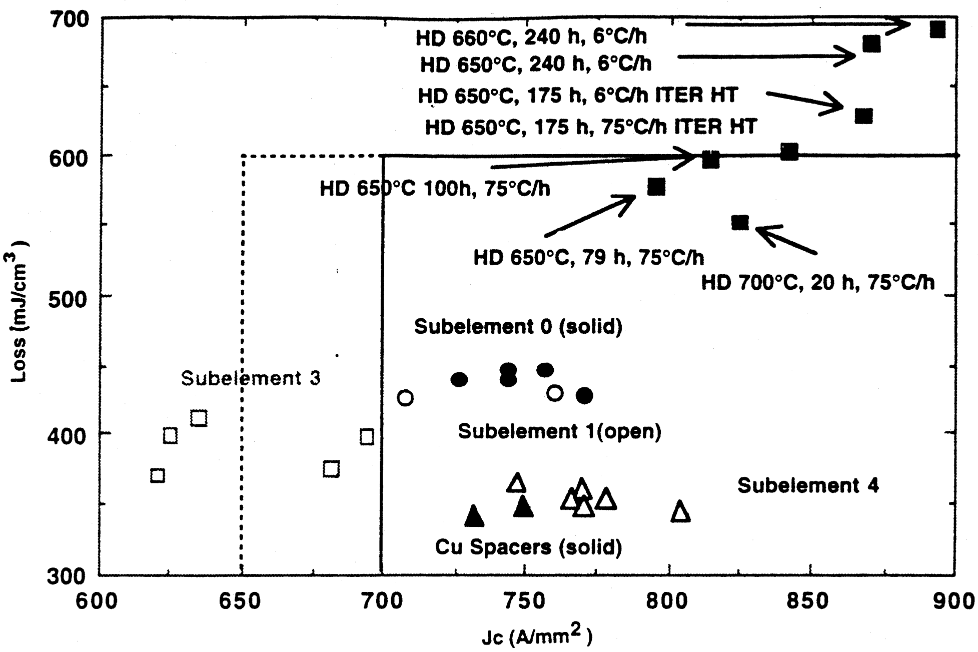

Fig. 5. Relationship between the J_c and the losses of "high density" strand after various heat treatments compared with that of standard material for Stage III of the ITER development program.

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

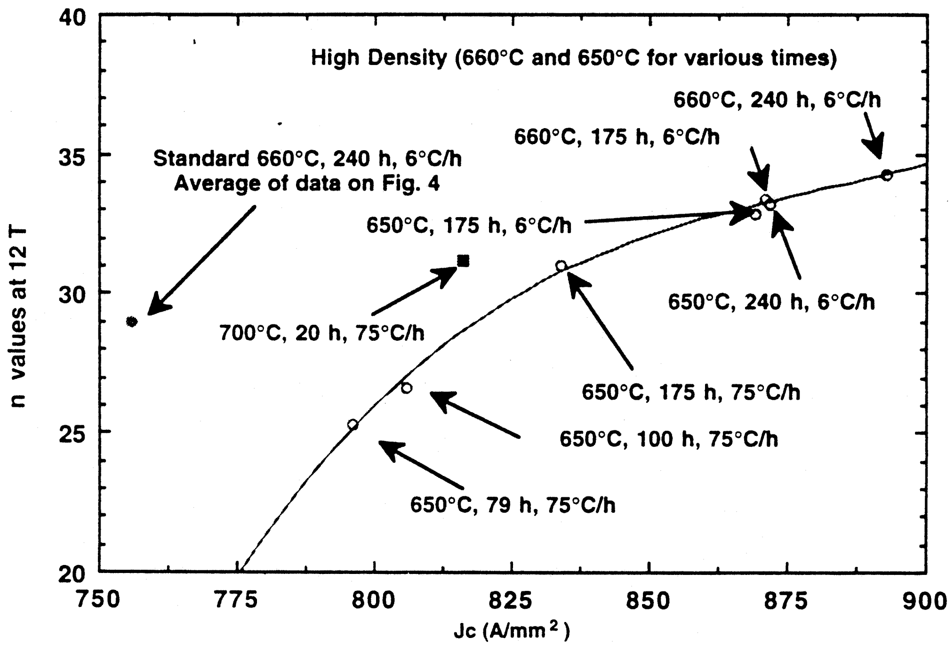

Fig. 6. Relationship between n value and J_c for "high density" strand after various heat treatments compared with that of standard material after 660°C for 240 h.

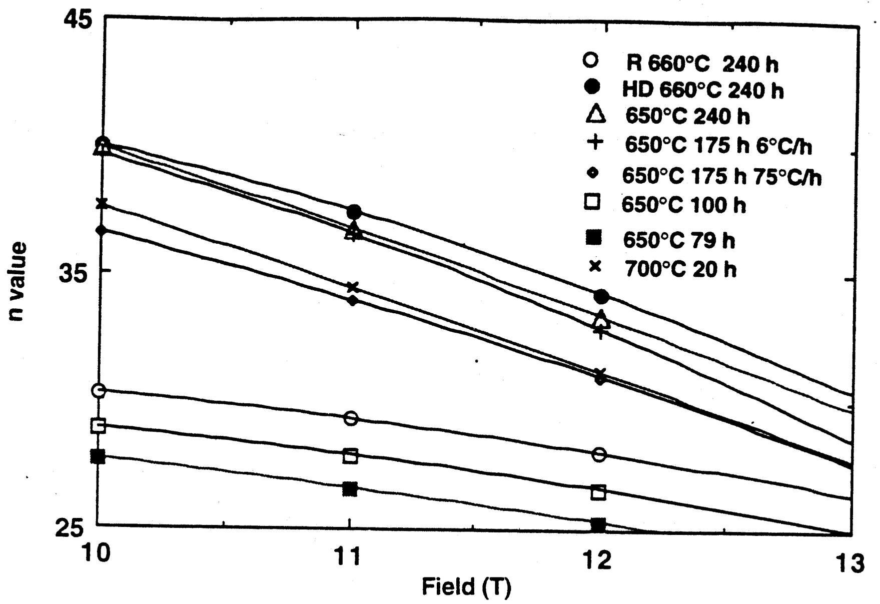

Fig. 7. Relationship between *n* value and field for "high density" strand after various heat treatments compared with that of standard material after 660°C for 240 h.

multifilamentary NbTi wire.[4,5]. In Nb_3Sn, 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 Nb_3Sn, 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 demonstate 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 Nb_3Sn 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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