

E. Gregory,<sup>1</sup> E. Gulko,<sup>1</sup> T. Pyon,<sup>1</sup> and L. F. Goodrich<sup>2</sup>

<sup>1</sup> IGC Advanced Superconductors, 1875 Thomaston Ave., Waterbury, CT 06704, USA

<sup>2</sup> National Institute of Standards and Technology, 325 Broadway, Boulder, CO 80303, USA

In papers [1&2], given a year ago, the results of round robin testing of International Thermonuclear Experimental Reactor (ITER) type Nb<sub>3</sub>Sn conductors have shown the variation of their J<sub>C</sub>'s with field. The IGC conductor, as heat treated to meet the ITER specification at 12 T, showed a steeper curve of J<sub>C</sub> against field than that of the other designs, suggesting a lower H<sub>C2</sub><sup>\*</sup> and T<sub>C</sub><sup>\*</sup>. Since the temperature and time of the heat treatment were restricted in order to ensure that the residual resistivity ratio (RRR) was not reduced by chromium diffusion, the slope of the curve had to be changed by means other than a simple heat treatment change. In this paper, a series of conductors with increased the H<sub>C2</sub><sup>\*</sup> & T<sub>C</sub><sup>\*</sup> and significantly reduced ac losses, are described.

## INTRODUCTION

An internal-tin Nb<sub>3</sub>Sn strand has been made meeting the ITER HP-1 specification [1-6]. An examination of some of the recent papers [1, 2], shows that this IGC conductor has a lower H<sub>C2</sub><sup>\*</sup> and a steeper J<sub>C</sub> versus field curve than certain other conductors. From very limited J<sub>C</sub> measurements at different temperatures in the range 4.0 K to 4.2 K and an extreme extrapolation suggested that the T<sub>C</sub><sup>\*</sup> of our conductor was somewhat lower than that of some other ITER conductors [3].

In this paper we report a method of altering the slope of the J<sub>C</sub>-versus-field curve, which is expected to result in the H<sub>C2</sub><sup>\*</sup> and T<sub>C</sub><sup>\*</sup> being raised to the values shown by other materials. An interesting fringe benefit of this change is that the losses at 12 T, as measured by the ITER criterion ( $\pm 3$  T in the non-Cu), appear to be lowered significantly. This is undoubtedly due, at least in part, to the lower J<sub>C</sub> at low field in the newer material.

## IMPROVEMENT IN PROPERTIES AT HIGH FIELDS.

Two recent papers by Bruzzone et al.[1, 2], clearly pointed out that a J<sub>C</sub>-versus-field curve of the regular IGC material was much steeper than that of the other round robin materials. It appeared therefore that the H<sub>C2</sub><sup>\*</sup> of this strand was lower than that of materials from other manufacturers.

Previous work [7, Fig. 2], suggested that the slope of the J<sub>C</sub> -vs.-field curve was steeper for the Nb 7.5 wt. % Ta than for material of a similar design containing Nb 1.3 wt. % Ti. It was not known whether this was due to compositional effects or small differences in heat treatment. Since the crossover in the curves was at 17 T, it was not originally of concern in the ITER work. Although the high field J<sub>C</sub> properties could be improved by more aggressive heat treatments, this was not possible for the Cr-plated ITER strand because the increased chromium diffusion would reduce the RRR of the stabilizing Cu.

### Material Preparation

Titanium additions to the matrix improve the high field properties of bronze processed material [8]. Such Ti additions to the Cu matrix improved the properties of our internal-tin material [9, 10], even when

<sup>†</sup> Contribution of the National Institute of Standards and Technology, not subject to copyright.

Ta was in the filaments [10]. A similar improvement can be obtained by adding Ti to the cores of the internal-tin material [11,12].

In an attempt to reduce the slope of the  $J_c$  vs. field and the  $T_c^*$  field curves, it was decided to add Ti to the Sn in three different subelement designs: regular ITER, "intermediate density" with a 4% increase in filament diameter, and "high density" with an additional 4% increase in filament diameter.

An alloy of Sn 2wt.% Ti was vacuum-melted and fabricated into 22.3 mm diameter bars by the Materials Preparation Center of the DOE Ames Laboratory, Ames, Iowa. Tubes from a previous subelement billet, #4 [4] were used for the "regular density" design. A tube from subelement billet 002 was used for the "intermediate density" design, and tubes from yet another subelement billet, 003, were used for the "high density" design. The Nb 7.5 wt. % Ta alloy rods for both subelement billets 002 and 003 were made from a single Teledyne WhaChang (TWC) heat which, while meeting the compositional specifications, appeared to contain above average impurity elements, in particular W, at approximately 250 parts in  $10^6$ .

The effect of the Ti addition to each subelement could easily be determined as the only change in each design was to replace the Sn/Cu alloy normally used in the subelements, with a Sn/Ti alloy.

### Critical current density

The six materials, three with Sn/Cu in the subelements and three with Sn/Ti, were processed into 0.81 mm diameter wire in the manner used to process ITER strand. The samples were heat treated for 240 h at 660°C after a ramp rate of 6°C/h. The cool down was carried out at a rate as close as possible to 25°C/h. Standard ITER holders were used for  $J_c$  testing and tests were carried out at MIT by IGC personnel. The loss measurements were made by R.B. Goldfarb at NIST.

Figure 1 shows the variation of  $J_c$ , (using non-Cu area) with magnetic field for the three designs of material listed above, with and without Ti added to the Sn core. "C" denotes the control materials with no Ti and "T" the Ti containing materials. "H," "I," and "R" are "high," "intermediate," and "regular" density. HW and LW stand for high and low tungsten (see above). The change in the slope with the Ti addition is the most noticeable feature. At higher fields, the  $J_c$ 's of the material with the Ti additions are superior to those of materials without such additions. This suggests that  $H_{c2}^*$  is improved by the addition of the Ti.

### Upper critical field ( $H_{c2}^*$ )

Figure 2 shows a Kramer plot of the two intermediate density materials compared with the other round robin materials extrapolated over a considerable range. These indicate an improvement in  $H_{c2}^*$  from ~23.7 T to ~29 T, resulting from the addition of Ti to the Sn. The corresponding non-Cu losses for these two materials were 590 mJ/cm<sup>3</sup> and 430 mJ/cm<sup>3</sup>. More high field data are necessary if an accurate determination of  $H_{c2}$  is to be obtained. Extrapolations of this type using Kramer plots may not be valid for Nb<sub>3</sub>Sn [13].

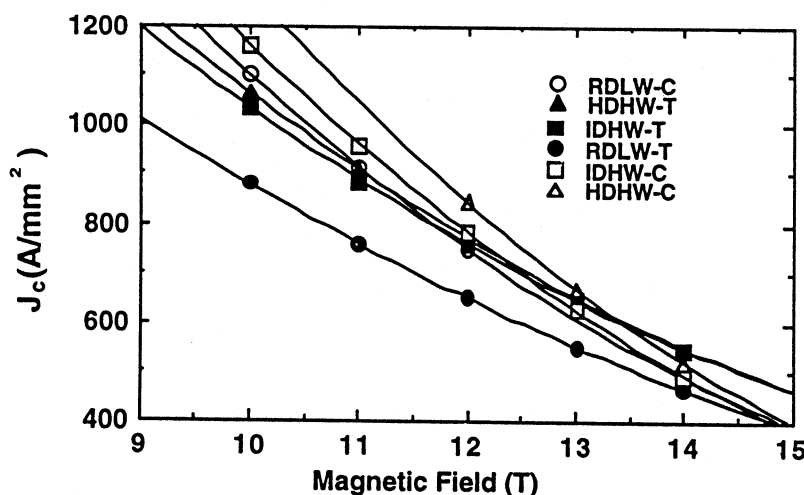


Figure 1.  $J_c$  vs field for materials containing Ti compared with non-Ti containing materials.

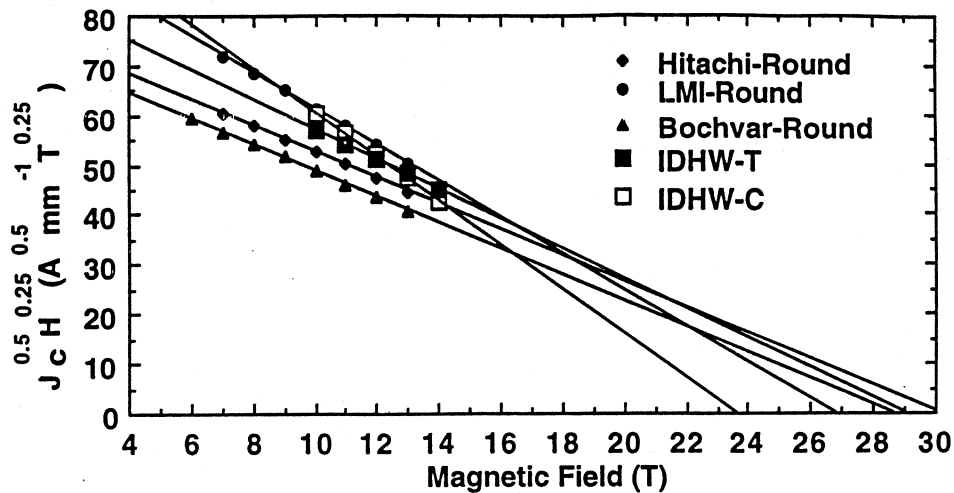


Figure 2. Kramer plots of Ti containing “intermediate density” material, IDHW-T, and non-Ti containing material, IDHW-C, and other round robin materials [1 & 2].

### Critical Temperature ( $T_c^*$ )

Based on the very limited data available from the benchmark tests, Bruzzone has suggested that  $T_c^*$  for our material was below 10 K at 12 T [2 & 3]. The two intermediate density materials, one with and one without Ti additions, were tested for  $I_c$  at a series of temperatures in the region 4.0 K to 5.0 K and again a long extrapolation of the data was made to determine  $T_c^*$ . The value obtained for the Ti-containing material was slightly above 11 K at 12 T and for the non-Ti containing material, Bruzzone's value of slightly below 10 K was confirmed. Again it is obviously necessary to obtain  $J_c$  data at higher temperatures.

### $n$ values

In Figure 3, the variation of  $n$  values with field is shown for the six materials described above. This figure indicates that the “high density” material shows the highest  $n$  values at all fields. The “intermediate density” material showed slightly lower values and the regular material the lowest.

The Ti-containing material has much higher values in all cases than the corresponding material without Ti. Also the Ti-containing material shows  $n$  values which do not fall off as rapidly with field as those of the materials without Ti.

## CONCLUSIONS

The addition of titanium into the ITER strand type design containing Nb 7.5 wt. % Ta filaments causes  $J_c$  to be lowered in the low field region and raised in the high field region. This is presumably due to an enhancement in the rate of formation of the A-15 compound with the concomitant increase in grain size. The decrease in  $J_c$  at low fields is a result of these larger grains whereas the improvement in the high field region is a result of the improved  $H_{c2}^*$ . The lower losses, as measured by the ITER criterion ( $\pm 3$  T in the non-Cu) follow from this lower  $J_c$  at low fields. The higher  $n$  values in the “higher density” materials are presumably due to the reduced spacing to diameter ratios. The value of  $J_c$  could be changed significantly if the heat treatment cycles were changed. The heat treatments used in this work did not give the optimum  $J_c$  as they were limited by the necessity of maintaining the RRR values required by the ITER specification in Cr-plated material.

## ACKNOWLEDGMENTS

This research is supported by the Department of Energy under subcontracts FC-A-395276 and FT-S-560409 from MIT and the Small Business Innovative Research, Grant numbers DE-FG02-91ER81153, DE-FG02-93ER81513 and DE-FG02-94ER81783. We particularly thank Dr. J. Minervini of the Plasma

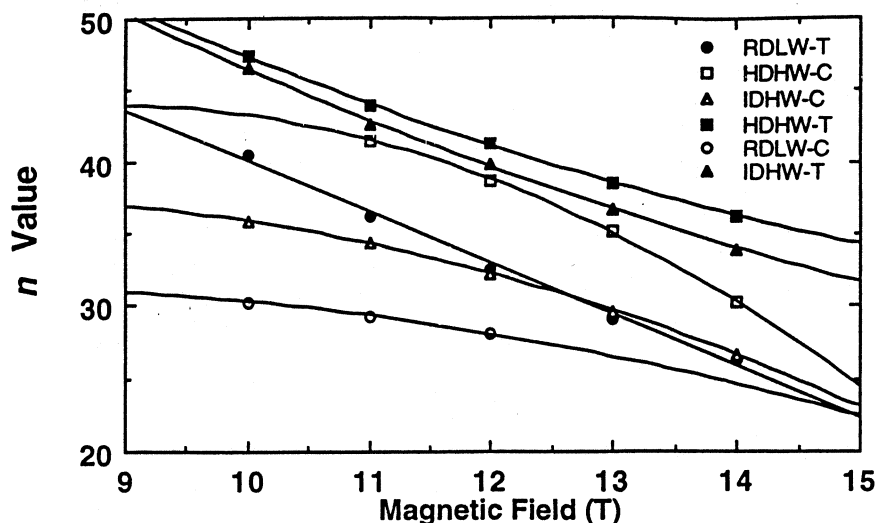


Figure 3. Variation of the  $n$  value with field.

Fusion Center (PFC) of MIT for his continuing financial and technical support of this work. We thank M. Takayasu of MIT (PFC), M. Suenaga of BNL and R.B Goldfarb of NIST for their help in heat treating and testing. Our thanks are also due to our colleagues at IGC who carried out much of the work described above, in particular, D. Birdsall, B. Boyle, R. Boyle M. Dormandy, and M. Vincenzi. Trade Names were mentioned in this paper to adequately specify this experimental study. In no case does such identification imply recommendation or endorsement by NIST

#### REFERENCES

1. Bruzzone, P., Mitchell, N., Steeves, M., Spadoni, M., Takahashi, Y and Sytnikov, V. E., Conductor fabrication for the ITER model coils, paper B27, 14th International Conference on Magnet Technology (MT-14) (1995), June 11-16, Tampere, Finland.
2. Bruzzone, P., ten Kate, H.H.J, Nishi, M. Shikov, A., Minervini, J., Takayasu M., Bench mark testing of Nb<sub>3</sub>Sn strands for the ITER model coil, Paper TU-PM2-17, CEC/ICMC (1995), Columbus, OH.
3. Bruzzone, P., private communication MT-14 (1995), June 11-16, Tampere, Finland.
4. Gregory, E., Gulko, E., Pyon, T. and Goodrich, L.F., Properties of an internal-tin Nb<sub>3</sub>Sn strand for the International Thermonuclear Experimental Reactor (ITER), Paper TU X3-2, CEC/ICMC (1995), Columbus OH.
5. Gulko, E., Pyon, T. and Gregory, E., The effect of heat treatment, tin concentration and filament size on the properties of internal-tin, Paper TU-PM21, CEC/ICMC (1995), Columbus OH.
6. Gregory, E., Gulko, E. and Pyon, T., Recent Improvements in the Properties of Internal-Tin Nb<sub>3</sub>Sn for Fusion Applications, to be presented at ASC (1996), August 25-30, Pittsburgh, PA.
7. Gregory, E., Ozeryansky, G.M. and Zeitlin, B.A. Improvement of the structure and properties of internal tin Nb<sub>3</sub>Sn conductors, Adv. in Cryo. Eng. (1992) Vol. 38B, pp. 579-586, eds. F.R. Fickett and R.P. Reed, Plenum Press NYC, NY Figure 2.
8. Tachikawa, K., Sekine H. and Iijima, Y., Composite-Processed Nb<sub>3</sub>Sn with Titanium addition to the matrix, J. Appl. Phys (1982), 53: 5354.
9. Ozeryansky, G. M., Gregory, E. and Zeitlin, B.A., An improved method of introducing additional alloying elements into Nb<sub>3</sub>Sn, IEEE Trans. on Applied Superconductivity (1993), Vol. 3, No. 1, pp. 1330-1333.
10. Pyon, T. and Gregory, E., Some effects of matrix Additions to internal tin processed multifilamentary Nb<sub>3</sub>Sn superconductors, IEEE Trans. on Applied Superconductivity (1995), Vol. 5, No. 2, pp. 1760-1763.
11. Suenaga, M., Klamut, C.J., Higuchi, N. and Kuroda, T., Properties of Ti alloyed multifilamentary Nb<sub>3</sub>Sn by internal-tin process, IEEE Trans. Magn. 21 (1985), 2, pp. 305-308.
12. Kuroda, T., Suenaga, M., Klamut, C.J. and Sabatini, R., Internal-tin processed Nb<sub>3</sub>Sn multifilamentary wires alloyed with Mg, Zn+Ni, and Ti through the Sn core, Adv. in Cryo Eng. (1986), Vol. 32, pp. 1011-1018, eds. R.P. Reed and A.F Clark.
13. Ekin, J., NIST, private communication, (1996)