To be presented at the 1990 Applied Superconductivity Conference, Snowmass Village, Colorado, Sept. 24-28, 1990

BNL -- 44556

MAGNETIZATION AND CRITICAL CURRENTS OF TIN-CORE MULTIFILAMENTARY Nb_3Sn CONDUCTORS

A. K. Ghosh and M. Suenaga
Brookhaven National Laboratory
Upton, New York 11973

BNL--44556

DE91 012241

Abstract

This paper presents critical current and magnetization data for some multifilamentary Nb_3Sn wires that have been produced by the *internal-tin method.* A comparison of magnetization and transport critical current measurements show that filament bridging during heat treatment is a common occurrence leading to effective filament diameters that are sometimes an order of magnitude larger than the geometrical filament size. At present, J_c's (in the non-copper region) greater than 1300 A/mm² at 10T have been achieved in some conductors, which also exhibit high losses. Low losses have only been seen in conductors with a high local ratio of niobium to copper. Also the use of (Nb-1%Ti) alloy instead of pure Nb helps to reduce low field loss and increase high field J_c. Measurements of the temperature dependence of hysteretic loss to 5T indicate that loss decreases linearly with increasing temperature.

Introduction

Nb_3Sn wire technology has progressed steadily during the last decade. Whereas some manufacturers have favored the traditional "bronze route", others, notably IGC Advanced Superconductors[1] (IGC) and Teledyne Wah Change, Albany[2] (TWCA) have relied on the internal-tin core method to produce high J_c wires at reasonable cost. Although both manufacturers use a tin core, their individual billet designs are unique and different. IGC's "Internal-Sn" method[3,4] uses a composite tube element of niobium filaments in a copper matrix filled with tin which is then cold drawn and restacked as subelements within a suitable barrier and copper stabilizer tube and then drawn to final wire size. TWCA's "modified jelly roll" (MJR)[5,6] approach uses an expanded metal mesh of niobium which is rolled up jelly roll fashion with copper sheet as the matrix material onto a core rod of tin. This composite is then drawn to form the subelement which is then similarly restacked within the stabilizer.

Over the years R&D has centered on producing high J_c (>1000A/mm² at 10T) wires with filament diameters (or thickness in the case of MJR) in the range of 1 to 5 microns. However, an early study of magnetization properties[7] showed that these wires had high hysteretic losses arising primarily from metallurgical bridging of the filaments in the subelement. These large magnetization could be characterized by an effective filament size which in some cases was an order of magnitude larger than the geometric size.

In this paper we review the magnetization and critical current properties of early wires produced by the tin-core method and present data on some recent wires that are being produced for high field dipole and fusion magnets and for laboratory research.

Experimental Details and Analysis

The wires used in this study were sui[illegible]y pre-reacted and then heat treated in the temperature range 650C-700C for periods of 48 to 150 hours depending on the wire and the manufacturing source. J_c optimization was not always done, hence the value quoted in the summary table is not necessarily the highest J_c achievable for that wire. The critical current, I_c, was measured in a different apparatus depending on the field of interest. In the low field range of 3T to 8T, I_c measurements are taken at an effective resistivity of 10^{14} Ω-m. The 8T to 10T measurements are reported for an I_c criterion of 10μv/m. Details of the measurement are given in ref. 8. The J_c quoted at 10T is for the non-copper region which includes the barrier.

Magnetization measurements are made using different techniques depending on the sample size. For long wire sampling (~10m) and fully finished Rutherford cables, an integration technique[7] is employed. For small wire lengths ~10 cm and variable temperature measurements a commercial SQUID magnetometer is used.[9] In this case the sample consists of a bundle of wires ~ 6 mm long.

Wire Description

In Table I, the relevant parameters of the Nb_3Sn samples that have been studied are listed. Included in the list, for comparison, are two bronze process wires: Sample #1 measured earlier at BNL[7] is a bronze route wire similar to that used in a dipole magnet at SACLAY[10] and #2 is a recent wire reported by Krauth[11] from Vacuumschmelze (VAC). Data for #2 wire has been taken from ref. 11. Wire similar to this was used to make a prototype LHC dipole magnet at CERN[12]. Sample #3 is an early IGC wire[13] with high I_c and #4 - #7 is a subset of 18 billets that IGC manufactured in the mid 80's to study the effect of local area ratio on d_{eff}. A report on this study is given in ref. 14. R. Goldfarb et. al.[15] has also reported on an independent study of loss for some of these samples. Samples #8 - #11 are recent wires made by IGC[16,17] with improved geometry to minimize d_{eff}. #12 is a prototype wire of a new process developed by IGC called the tin tube process (TTS)[18]. Samples #13-#14 were made by TWCA for a high field magnet being developed at BNL and reported elsewhere in these proceedings[19]. #15 is a low loss version developed for the US-DPC coil being built by MIT[20]. The table contains references in which the strand cross sections are depicted.

Effective Filament Diameter d_{eff}

A typical magnetization hysteresis loop is shown in Fig. 1. To characterize the magnetization properties of the wire it is convenient to define an effective filament diameter d_{eff} from the width of the magnetization loop[7] at a field larger than the full penetration field. This width $2\mu_oM$ can be written as

$$2\mu_oM(H) = 2\mu_o\ (2/3\pi)\ \lambda J_c(H)d_{eff} \qquad (1)$$

where λ is the area fraction of the non-copper region and $J_c(H)$ is the critical current density of this same area. In eq. (1) $2\mu_oM$ is in units of Tesla, J_c in A/m² and d_{eff} in meters. If A is the area of the wire cross section and I_c is the critical current measured at the field H, then

$$d_{eff} = (2\mu_oMA)/[2\mu_o(2/3\pi)\ I_c(H)] \qquad (2)$$

When using eq. (2) to calculate d_{eff}, $I_c(H)$ should be corrected for self field[7] especially if H is low. Typically d_{eff} is calculated at 5T using the measured self field corrected I_c at the same field. However, when only high field data are available, the I_c at low field is estimated from the J_c (H) behavior as shown in subsequent sections. If d_{eff} is equal to the geometrical filament diameter, d, then the filaments behave as if uncoupled and as expected from the critical state model. The hysteresis loss Q_H, is evaluated from the area under the M-H loop for a given field sweep, eg. ± 5T. Q_H would depend not only on d_{eff} and I_c(5T), but would be influenced by the barrier properties as well as the J_c behavior at low fields. This as shown later depends on the alloy composition used.

* Work done under the auspices of DOE

Manuscript received September 24, 1990.

Table I. Summary of Conductor Parameters and Properties

Sample #	Process/Alloy	Wire Dia. mm	Cu/NCu	Diff. Barrier Comp.	Diff. Barrier %	Cu/Nb ratio	%SC.	Fil. Dia. d, µm	J_c (10T) a/mm^2	deff µm	J_c (Nb_3Sn) A/mm^2	Reference #
See Footnotes					a	b	d		c	f	e	
1. (MCA)	Bronze	0.68	0.50	Ta	9	1.8	23.0	4.0	600	4	1890	10
2. (VAC)	Bronze/nB-7.5%Ta	0.92	0.62	Ta		1.5	24:0	3.0	800	7	2415	11
3. (IGC)	INT-Sn/Nb	0.68	1.00	Ta	13	1.2	28.0	2.0	1024	20	2650	13
4. (IGC-5901	INT-Sn/Nb	0.68	0.92	Ta	14	1.5	22.5	5.1	890	17	2866	14
5. (IGC-5901)	INT-Sn/Nb	0.68	0.92	Ta	14	1.5	22.5	2.8	930	11	2995	14
6. (IGC-5901)	INT-Sn/Nb	0.68	0.92	Ta	14	1.8	18.6	4.6	840	11	3273	14
7. (IGC-5901)	INT-Sn/Nb	0.68	0.92	Ta	14	1.8	18.6	2.6	740	5	2881	14
8. (IGC-5956)	INT-Sn/Nb	0.78	1.08	Ta	9	1.8	19.7	3.5	650	7.1	2391	16
9. (IGC-5953)	INT-Sn/Nb	0.83	1.01	Ta	24	1.2	20.0	3.8	850	12.8	3080	
10. (IGC-5984)	INT-Sn/Nb-1%Ti	0.84	1.05	Ta	18	1.0	18.7	4.2	940	14.6	3643	17
11. (IGC-5984)	INT-Sn/Nb-1%Ti	0.82	0.85	Nb/Cu	15	1.0	17.5	4.2	925	52	3830	17
12. (IGC)	Sn-Ring/Nb-1%Ti	0.52	1.27	Ta	10	0.8	31.5	4.0	1550	25	3554	18
13. (TWCA-1110)	MJR-Sn/Nb-1%Ti	0.84	1.8	V/Nb	15	0.8	33.5	2*7(4)	1240	80	2682	19
14. (TWCA-1072)	MJR-Sn/Nb-1%Ti	0.84	1.8	V/Nb	13	1.5	25.0	2*7(4)	840	24	2435	19
15. (TWCA-1094)	MJR-Sn/Nb-1%Ti	0.78	1.15	V	13	1.7	23.0	4*8(6)	740	17	2331	20

a : percentage area of barrier in the region excluding the stabilizer Cu
b : local area ratio in the Cu (CuSn)- Nb composite
c : J_c for non-copper region. J_c at 10T defined for 10µV/m criterion
d : percentage area of Nb (or Nb-1%Ti) as filaments in the non-Cu region
e : J_c @10T normalized for Nb_3Sn assuming 37% volume expansion during annealing
f : effective filament diameter as calculated from magnetization data using eq. (2)

Results and Discussion

In this section we address the question of J_c and d_{eff}. We also show that the low field $J_c(H)$ behavior is more favorable for the Nb-1%Ti alloy than for pure Nb. Data on the temperature dependence of the hysteretic loss Q_H is presented and the effect of the barrier material is discussed in relation to d_{eff} and low field loss.

Critical Currents and d_{eff}

Table I summarizes the J_c and magnetization data for the samples listed. In almost every case except sample #1, d_{eff} is greater than d indicating that filament bridging is a common occurrence even in bronze processed wire. An example of filament bridging is shown in Fig. 2. Fig. 3 is a plot of d_{eff} versus filament size for all the samples except #11 and #13. Most of the data lie between d_{eff} = 2 to 4 times d. Clearly, the least value of d_{eff} is for a conductor with the smallest filaments and the highest local area ratio. This is essentially the same conclusion drawn from the earlier study[14] in which 18 IGC billets were measured to study the effect of local ratio and filament size on d_{eff}.

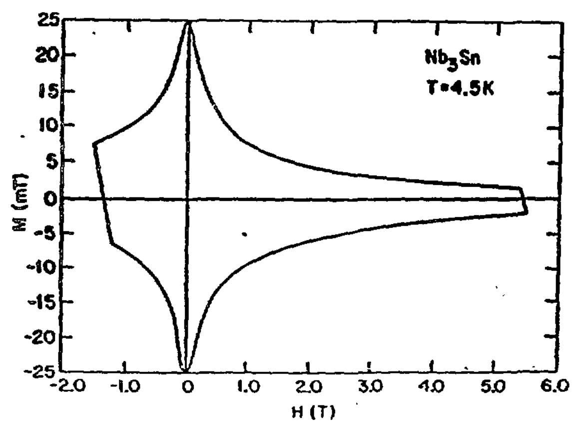

Fig. 1 A typical magnetization measurement. Data shown for sample #8:

Another interesting plot is that of d_{eff} versus critical current density J_c at 10T, which is shown in Fig. 4. Here one finds that d_{eff} increases as the wire J_c increases, which means that the magnetization or loss increases non-linearly with J_c. For the IGC "Internal-Sn" process a J_c of ~1000A/mm^2 can be achieved for a d_{eff} ~ 14 µm. The MJR-Sn process seems to have higher losses than the "internal-Sn" wires billet for comparable J_c. However, recently TWCA[21] has successfully fabricated a low loss wire with a J_c of 1050A/mm^2.

At present the highest J_c measured in an internal tin wire is for an experimental billet made by IGC with 91 subelements containing 1µm filaments with a niobium area fraction of ~ 39% of the non-copper region. The local ratio was ~ 0.8. This wire had a J_c > 2000A/mm^2 at 10T. However, the wire had a high loss with the magnetization consistent with the subelements acting as a single large filament of diameter ~ 40 µm. Optimized TWCA-MJR wires[6] similar to sample #13 have been reported to have J_c in excess of 1500 A/mm^2. These wires too have very high losses. Interestingly enough, a bronze processed wire with local ratio of 0.8 was also measured to have a high loss with d_{eff}/d~10. It appears that using local area ratios of less than 1.0 usually produces wires with high loss.

a) b)

Fig. 2 a) Filament bridging and b) the filament surface of an internal-Sn wire, (Sample #10). No "sausaging" observed along the filament length.

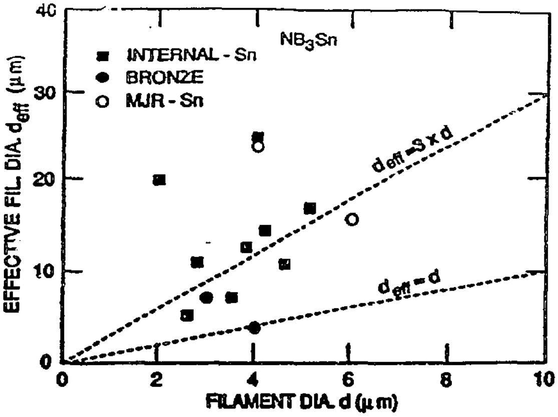

Fig. 3 Plot of d_{eff} versus filament diameter, d.

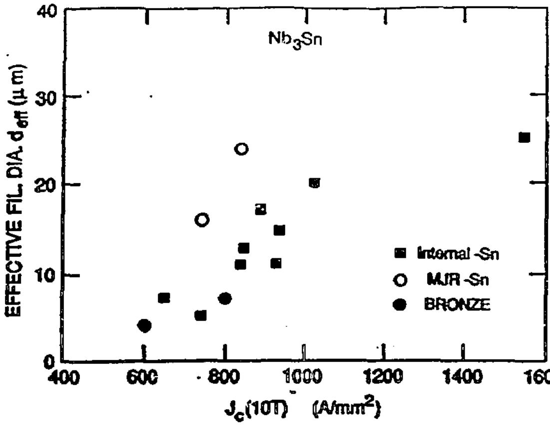

Fig. 4 Plot of d_{eff} versus the critical current density, J_c (10T).

$(Nb-1\%Ti)_3Sn$ versus Nb_3Sn

It is well know from studies of bronze processed wires[22], that the high field J_c of Nb_3Sn alloyed with 1%Ti has a shallower J_c-H slope than wires made with pure Nb. This is also evident from Table I when the J_c of Nb_3Sn is calculated from the known area fraction of the superconductor assuming complete reaction. The highest J_c ~3700 A/mm^2 at 10T is achieved in wires using Nb-1%Ti alloy. To map this difference as a function of field the ratio $J_c(H)/J_c(8T)$ versus H is plotted in Fig. 5 for samples #9 and #10 which have the same J_c at 8T. In this plot, the J_c at fields < 5T is obtained from the magnetization width and the calculated d_{eff}. From this figure one expects that the low field loss would be smaller for the wires with Nb-1%Ti alloy. In fact the ± 3T loss for sample #10 was ~20% lower than that for the #9 conductor. In extrapolating J_c data from 8T to low fields, for the purpose of comparing magnetization and critical current information, the values given in Table II were used. These are average values for the two classes of wires. Conductors made with Nb-7.5%Ta alloy is expected to behave similarly to the Nb-1%Ti alloy conductor.

Table II. $J_c(H)/J_c(8T)$ Ratio

H(T)	Nb_3Sn	$(Nb-1\%Ti)_3Sn$
3	4.20	3.10
5	2.20	1.85
10	0.59	0.69

Diffusion Barrier

The choice of the barrier material is a vexing problem for the wire manufacturers. For economic as well as for drawability reasons, if a niobium or a combination of niobium/copper is used then the formation of Nb_3Sn at the copper-niobium interface produces a large magnetization at all fields[7] leading to a high d_{eff} (sample #11). Use of the traditional tantalum barrier, although suitable from the loss point of view, has its own problems of drawability and piece length in the manufacture of the wire. TWCA has had a great

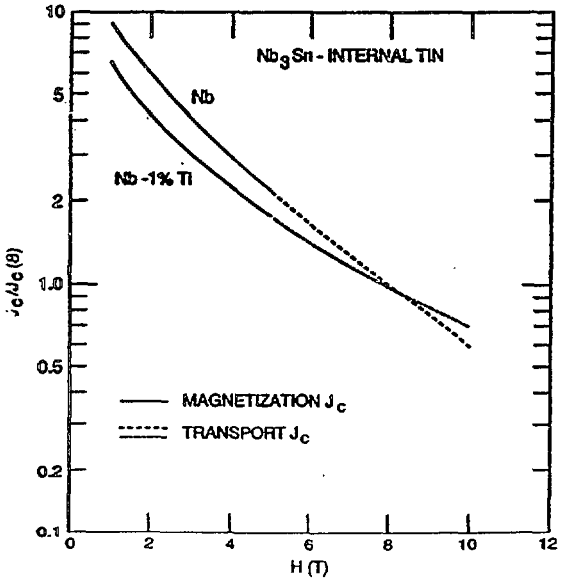

Fig. 5 The normalized $J_c(H)$ behavior of Nb_3Sn and $(Nb-1\%Ti)_3Sn$. Data given for samples #9 and #10.

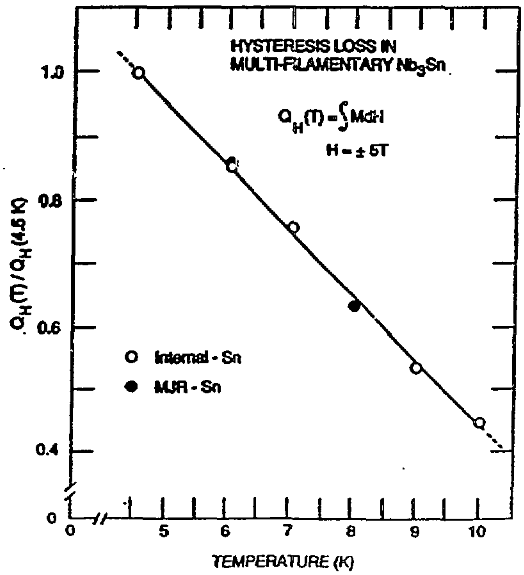

Fig. 6 The temperature dependence of Q_H (±5T). The loss is normalized to the value at 4.5K.

deal of success in utilizing vanadium or a niobium/vanadium barrier in their wire production[6]. Use of this barrier does not lead to increased losses except at fields where niobium is superconducting i. e. less than 0.6T.

Temperature Dependence of Q_H

The temperature dependence of $Q_H(\pm 5T)$ was measured for samples #8 and #15. Conductor #8 had a 4.5K loss of 120 mJ/cm^3 whereas #15 had a loss of 188 mJ/cm^3. Fig. 6 is a plot of Q_H versus T, where the loss is normalized to its value at 4.5K. The limited data show that Q_H scales linearly with temperature for both Nb_3Sn and $(Nb\text{-}1\%Ti)_3Sn$ conductors. The linear dependence can be written as

$$Q_H(T)/Q_H(4.5K) = 1.47 - 0.103T \qquad (3)$$

Conclusions

The choice of the conductor design depends largely on the requirements of the high field device. Clearly, for device operation at 10-12T, the alloy superconductor Nb-1%Ti (or Nb-7.5%Ta) guarantees a higher current density than pure Nb. Additionally, these alloys have the added advantage of lowering the overall hysteretic loss.

In developing conductors for magnet operations a ~10T, the overall J_c requirement has to be traded with acceptable loss or low field magnetization. At present this imposes some restrictions on the choice of J_c. Where the application is such that loss is not a major concern, conductors like #12 and #13 are suitable to build high field magnets. With a high J_c ~1500 A/mm^2, the wire can have a sufficient copper content for conductor stability and quench protection, without appreciably sacrificing the current carrying capacity. In utilizing bronze process wire like #2, the stabilizer content has to be reduced significantly to achieve a high I_c. This may compromise the quench protection of high inductance dipole magnets (like the LHC magnet), since the current density in the copper can become unacceptable large thereby requiring active energy extraction during a quench to prevent magnet burnout.

For accelerator magnets where the low field magnetization properties are important for machine operation, Fig. 4 would indicate that at present an acceptable conductor would have a moderate J_c (10T) ~ 1000 A/mm^2 and a d_{eff} ~ 14 µm and a barrier of either tantalum or vanadium. It is possible that further refinements of the billet geometry could decrease d_{eff} to the range of 7-9 µm primarily by reducing d~2 µm.

To achieve a low loss conductor, the J_c is restricted to values < 800A/mm^2 with local area ratio preferably ~ 1.5, with filament diameters ~ 2 µm. This wire requires the use of a tantalum or a vanadium barrier.

Acknowledgements

The authors would like to thank W. B. Sampson for numerous discussions. Also, the authors express their appreciation to the commercial multifilament Nb_3Sn wire manufactures notably IGC Advanced Superconductor and TWCA, who contributed samples for this study. The expert technical assistance of F. Perez, J. D'Ambra, E. Sperry and R. Sabitini were greatly appreciated.

References

1. IGC Advanced Superconductors, Waterbury, CT., 06704.
2. Teledyne Wah Chang Albany, Albany, OR, 97321.
3. R. E. Schwall, G. M. Ozeryansky, D. W. Hazelton, S. F. Logan and R. M. Rose, "Properties and Performance of High Current Density Sn-Core Process MF Nb_3Sn", IEEE Trans. on Magnetics, 19, 1135, (1983).
4. B. A. Zeitlin, G. M. Ozeryansky and K. Hemachalam, "An Overview of the IGC Internal Tin Nb_3Sn Conductor", IEEE Trans. on Magnetics, 21, 293, (1985).
5. W. K. McDonald, C. W. Curtis, R. M. Scanlan, D. C. Larbalestier, K. Marken and D. B. Smathers, Manufacture and Evaluation of Nb_3Sn Conductors Fabricated by the MJR Method", IEEE Trans. on Magnetics, 19, 1124, (1983).
6. D. Smathers, P. O'Larey, M. Siddall and W. McDonald, "Status of the Superconductor Development Program at Teledyne Wah Chang Albany", Adv. in Cryo. Engr.-Matr., Vol. 34 (Plenum Press, NY), p. 515, (1988).
7. A. K. Ghosh, K. E. Robins and W. B. Sampson, "Magnetization Measurements on Multifilamentary Nb_3Sn and NbTi Conductors", IEEE. Trans. on Magnetics, 21, 328, (1985).
8. M. Suenaga, K. Tsuchiya, N. Higuchi and K. Tachikawa, "Superconducting Critical-Current Densities of Commercial Multifilamentary $Nb_3Sn(Ti)$ Wires Made by the Bronze Process", Cryogenics, 25, 123, (1985).
9. MPMS SQUID MAGNETOMETER, Quantum Design, San Diego, CA, 92121.
10. J. Perot, "Construction and Test of a Synchrotron Dipole Model Using Nb_3Sn Cable", IEEE. Trans. on Magnetics, 19, 1378, (1983).
11. H. Krauth, "Development and Large Scale Production of NbTi and Nb_3Sn Conductors for Beam Line and Detector Magnets", Proc. of the 2nd Annual Int. Industrial Symp. on the Super Collider, Miami Beach, Florida, Mar. 14-16, (1990), (to be published).
12. S. Wenger, F. Zerobin and A. Ashner, "Towards a 1 m long High Field Nb_3Sn Magnet of the ELIN -CERN collaboration for the LHC-Project", IEEE Trans. on Magnetics, 25, 1636, (1989).
13. N. Higuchi, K. Tsuchiya, C. J. Klamut and M. Suenaga, "Superconducting Properties of Nb_3Sn Multifilamentary Wires Fabricated by Internal Tin Process, "Adv. in Cryo. Eng. Matr., vol. 30, 739, (1982).
14. A. K. Ghosh and W. B. Sampson, "Magnetization Studies of Multifilamentary Nb_3Sn Wires", Brookhaven National Laboratory Report, BNL- 41160, (1988)
15. R. B. Goldfarb and J. W. Ekin, "Hysteresis Losses in Fine-Filament Internal-Tin Superconductors", Cryogenics, 26, 478, (1986).
16. H. C. Kanithi, L. R. Motowidlo, G. M. Ozeryansky, D. W. Hazelton and B. A. Zeitlin, "Low Loss and High Current Nb_3Sn Conductors Made by the Internal Tin Method", IEEE Trans. on Magnetics, 25, 2204, (1989).
17. E. Gregory, G. M. Ozeryansky, R. M. Schaedler, H. C. Kanithi and B. A. Zeitlin, "An Internal Tin Conductor with Nb-1 wt % Ti filaments", Adv. in Cryo. Eng. Matr., Vol 36, (Plenum Press, NY), p. 147, (1990).
18. G. M. Ozeryansky, E. Gregory, "A New Internal Tin Nb_3Sn Conductor Made by a Novel Manufacturing Process", paper LHP-2, ASC Conference, Snowmass, CO., (1990).
19. R. McCluskey, K. Robins and W. B. Sampson, "A Nb_3Sn High Field Dipole", ASC Conference, Snowmass, CO., (1990).
20. D. E. Smathers, P. M. O'Larey, M. M. Steeves and M. D. Hoenig, "Production of Tin Core Modified Jelly-Roll Cable for the M.I.T. Multipurpose Coil", IEEE Trans. on Magnetics, 24, 1131, (1988).
21. D. Smathers, private communication.
22. M. Suenaga, S. Okuda, R. Sabitini, K. Itoh and T. S. Luhman, "Superconducting Properties of $(Nb,Ti)_3Sn$ Wires Fabricated by the Bronze Process", Adv. in Cryo. Eng. Matr. Vol. 28, (Plenum Press, NY), p. 379, (1982).