Comparative Measurements of ITER Nb$_3$Sn Strands Between Two Laboratories

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***Abstract*—ITER Nb$_3$Sn strand quality verification tests require large quantities of precise measurements. Therefore, regular cross-checking between testing laboratories is critically important. In this paper, we present results from a cross-checking test of 140 samples between the National High Magnetic Field Laboratory, USA, and the University of Twente, The Netherlands. The tests comprise measurements at 4.2 K on critical current, residual resistance ratio, and hysteresis loss, while at room temperature the chromium layer thickness, Cu/non-Cu ratio, filament twist pitch, and diameter were determined. Our results show very good agreement between the two laboratories. The reasons for small random discrepancies are discussed.**

***Index Terms*—Critical current, hysteresis loss, Nb$_3$Sn, residual resistance ratio (RRR).**

I. INTRODUCTION

LARGE superconducting magnets such as those for the ITER International Organization require commercial production of a large quantity of Nb$_3$Sn strands [1]. It demands accurate and cost-effective quality assurance measurements to assure reliable operation of the magnets. While testing superconducting strands is very important for large superconducting magnet projects in general [2]–[5], it is particularly crucial for ITER Nb$_3$Sn strands, which are supplied by multiple strand manufacturers. Therefore, a verification test program is implemented, involving a number of laboratories worldwide [6]–[17].

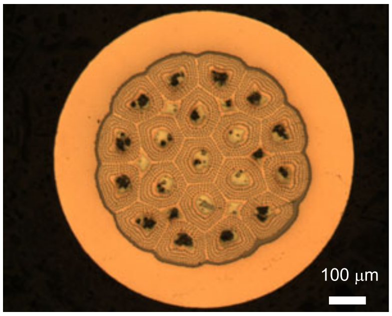

Fig. 1. Cross section of an unreacted Nb$_3$Sn strand used in this experiment.

Historically, Nb$_3$Sn wire manufacturers and various research laboratories use slightly different testing protocols. Therefore, the ITER International Organization organized benchmarking and annual cross-checking tests to be performed at each testing laboratory, which carries out heat treatment and quality property-verification tests of a few samples [6], [17]. These tests are typically performed on samples cut from a single-piece length of Nb$_3$Sn wire, assuming sufficient quality and homogeneity of the properties along the wire length. Due to the small number of test samples for each participating laboratory, analysis of the statistical difference between laboratories is difficult. This paper presents the results of a special set of cross-checking measurements of a large number of production samples, which is considerably more than the number of ITER routine cross-checking samples. In this experiment, samples from 140 billets made by one manufacturer are prepared, heat treated, and tested by both the National High Magnetic Field Laboratory (NHMFL), USA, and the University of Twente (UT) in a production testing mode. We compare the test results of critical current (I_c), residual resistance ratio (RRR), hysteresis loss (Q_{hyst}), diameter, chromium-coating thickness, twist pitch, and copper to non-copper ratio between two laboratories. The relatively large number of tests allows us to perform statistical analysis and to identify the significance of the difference or lack thereof between the two sets of data.

II. EXPERIMENTAL METHODS

Nb$_3$Sn strands used in this experiment are designed for the ITER Toroidal Field coils and manufactured by the internal-tin process by Luvata (Waterbury, CT, USA). A cross-section of an unreacted strand is shown in Fig. 1. A 20-m long wire is cut from each of the 140 billets for testing. NHMFL and

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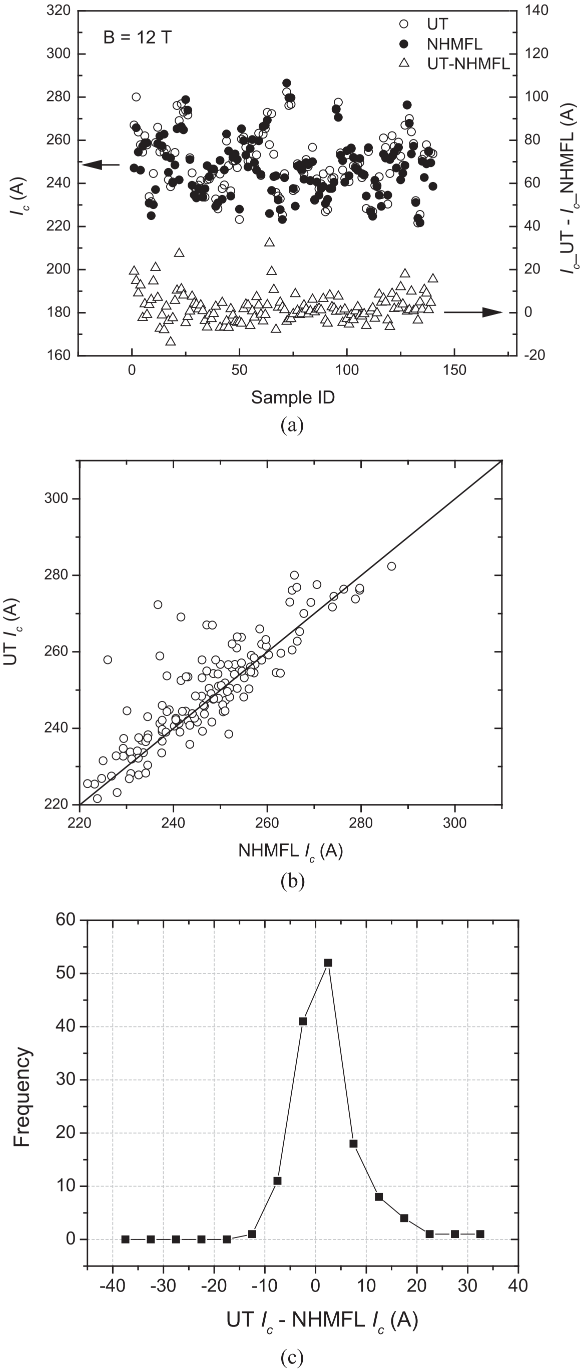
TABLE I
TEST METHODS USED BY NHMFL AND UT

	# of tests	NHMFL	UT
Heat treatment	140	In argon, sheathed K-type thermocouple, ITER schedule B	In vacuum, N-type thermocouple, ITER schedule B
I_c	140	ITER barrel, no handling after HT, 25 and 50 cm taps	ITER barrel, no handling after HT, 25- and 50-cm taps
RRR	80	150-mm straight sample, natural warming to 20 K	100-mm straight sample, natural warming to 20 K
Q_{hyst}	40	VSM, 7-turn, 4-mm-diameter coil	Magnetometer, 14-turn, 40-mm-diameter coil, 2-m wire length
Cr thickness	80	Light microscopy	Etching, weigh
Cu/non-Cu	80	Light microscopy	Etching, weigh
Twist Pitch	140	Etch, incline angle	Etch, incline angle
Diameter	80	Digital micrometer	Digital micrometer



(a)

(b)

(c)

Fig. 2. I_c measured at 4.2-K 12-T, comparison between NHMFL and UT for 140 samples. (a) Left vertical axis is for I_c measured by NHMFL and UT, the right vertical axis is for the I_c difference between UT and NHMFL. (b) UT I_c versus NHMFL I_c, the diagonal line indicates an ideal correlation between the two sets of data. (c) Histogram of UT I_c−NHMFL I_c. Curve centers approximately at zero indicating statistical consistency between UT and NHMFL data.

UT, each performed independent heat treatment, liquid-helium temperature testing, and room-temperature testing.

Reaction heat treatment at both laboratories were performed following the ITER heat treatment schedule B:

1) 210 °C for 50 h;
2) 340 °C for 25 h;
3) 450 °C for 25 h;
4) 575 °C for 100 h;
5) 650 °C for 100 h;
6) cool to 500 °C + furnace cool;
7) The ramp rate = 5 °C/h for all ramps.

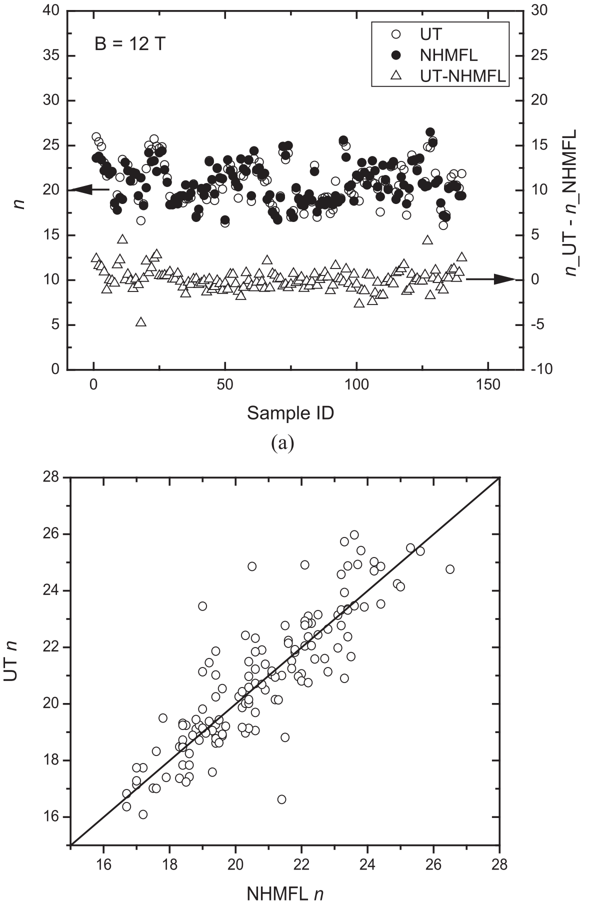
At NHMFL, heat treatment of total 140 samples was done in six consecutive batches in the flowing Ar gas. I_c, RRR, and Q_{hyst} samples from the same strand were always heat treated in the same batch. The heat treatment at UT was completed in one batch in vacuum. Some details of the measurement techniques adopted at the NHMFL are given in [7], and of the UT test techniques in [11] and [12]. For ease of comparison, a brief description of test methods used by each laboratory is listed in Table I, which also contains the number of samples for each test. The RRR of ITER Nb_3Sn strand is defined as its resistance ratio between 273 and 20 K. Both I_c and n were measured at 11.5, 12.0, and 12.5 T, but only 12.0 T data are presented in this paper. I_c self-field corrections were not applied.

III. RESULTS AND DISCUSSIONS

A comparison of I_c measured by NHMFL and UT is shown in Fig. 2(a). It is evident that these two sets of data are in good agreement with one another. The I_c difference between UT and NHFML, I_c_UT − I_c_NHMFL (ΔI_c), is also plotted for each sample in this figure. The ΔI_c varies randomly around zero, and the variation of ΔI_c from billet to billet is somewhat smaller than the variation of I_c. Fig. 2(b) shows I_c measured by UT against I_c measured by NHFML. Data scatter randomly around the diagonal line which corresponds to a perfect UT–NHMFL agreement. A histogram of ΔI_c is presented in Fig. 2(c), which centers approximately at zero, and shows that there is no significant systematic difference between UT and NHMFL I_c data. It is noted, however, that a few ΔI_c values are as large as 30 A. Since the uncertainty in I_c measurement is typically only about 3 A [6], and there is no evidence of discrepancy in the heat treatment which would cause a systematic difference between

TABLE II
SUMMARY OF TEST RESULTS

Test	NHMFL		UT		UT–NHMFL	
	Mean	Standard deviation	Mean	Standard deviation	Mean	Standard deviation
I_c (12 T) (A)	247.4	13.2	249.7	13.6	2.3	6.9
n	20.8	2.1	20.8	2.4	0.1	1.2
RRR	154.4	13	154.8	11.3	0.4	9.0
$Q_{\rm hyst}$ $({\rm kJ/m^3})$	350.0	50.8	341.5	39.1	−8.4	43.6
Cr thickness (μm)	1.3	0.3	1.3	0.3	0.0	0.2
Cu/non-Cu	0.916	0.012	0.902	0.014	−0.013	0.016
Twist Pitch (mm)	15.4	0.8	16.8	1.2	1.5	1.5
Diameter (mm)	0.819	0.001	0.821	0.001	0.003	0.001



(a)

(b)

Fig. 3. n value measured at 4.2-K 12-T, comparison between NHMFL and UT for 140 samples. (a) Left vertical axis is for n measured by NHMFL and UT, the right vertical axis is for the n difference between UT and NHMFL. (b) UT n versus NHMFL n, the diagonal line indicates an ideal correlation between the two sets of data.

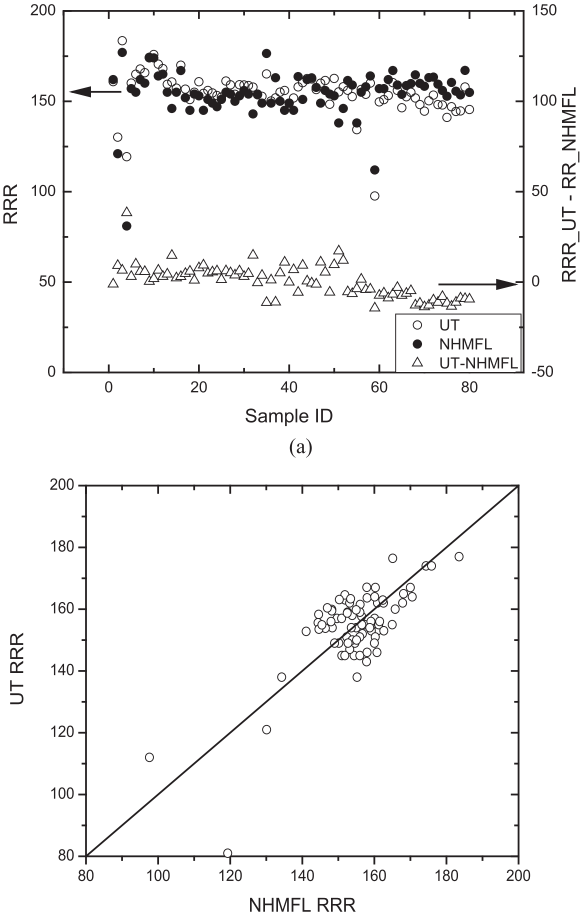


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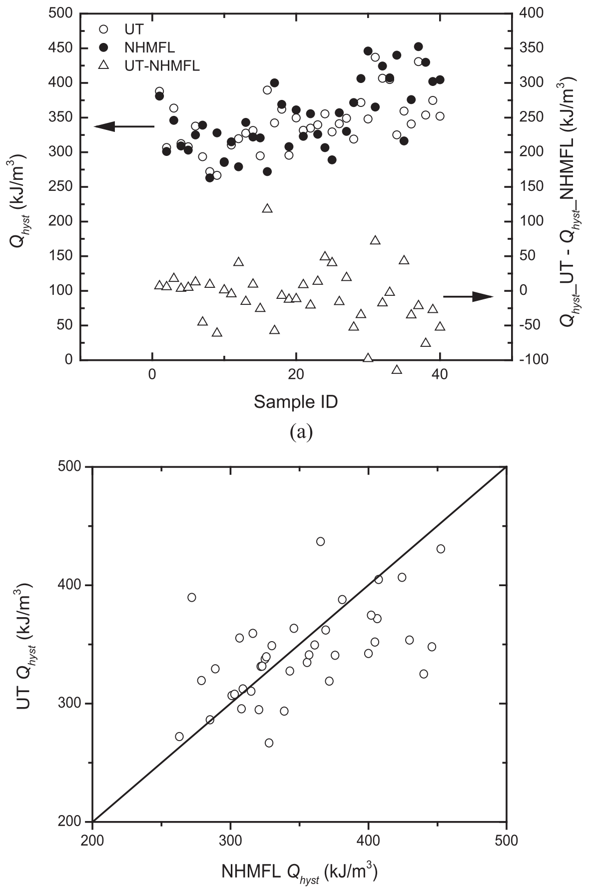
Fig. 4. RRR comparison between NHMFL and UT for 80 samples. (a) Left vertical axis is for RRR measured by NHMFL and UT, the right vertical axis is for the RRR difference between UT and NHMFL. (b) UT RRR versus NHMFL RRR, the diagonal line indicates an ideal correlation between the two sets of data.

UT and NHMFL I_c, and I_c is not particularly sensitive to the heat-treatment temperature within +/− 10 °C [18], we suspect that the large ΔI_c is due to the appreciable property variation within the 20-m sampling length for each billet. This hypothesis is supported by the fact that I_c variation within a billet can be as much as 30 A for wires made by multiple manufacturers including Luvata [19].

Similar plots of n value, RRR, and $Q_{\rm hyst}$ comparisons are shown in Figs. 3–5, respectively. Similar to the case with I_c, the agreement between UT and NHMFL is good. The relatively larger variations in Δn, ΔRRR, and $\Delta Q_{\rm hyst}$ cannot be explained by the uncertainty of measurement techniques which is about 1, 1, and 3 kJ/m^3 for Δn, ΔRRR, and $\Delta Q_{\rm hyst}$, respectively [20]. Again, they might be attributed to the nonuniformity of



(a)

(b)

Fig. 5. Q_{hyst} comparison between NHMFL and UT for 40 samples. (a) Left vertical axis is for Q_{hyst}, measured by NHMFL and UT, the right vertical axis is for the Q_{hyst}, difference between UT and NHMFL. (b) UT Q_{hyst} versus NHMFL Q_{hyst}, the diagonal line indicates an ideal correlation between the two sets of data.

properties within 20 m of sample for each billet, and are supported by observed variations in Δn, ΔRRR, and ΔQ_{hyst} within one billet [19]. Finally, a summary of results and statistics for all tests including room-temperature test is shown in Table II. The difference in mean values between NHMFL and UT data is small as compared with random data scattering quantified by standard deviation, except for the diameter measurement, which requires further investigation.

4. CONCLUSION

NHMFL and UT conducted cross-checking measurements of 140 internal-tin Nb_3Sn wires. Samples were heat treated and tested in each laboratory separately. Comparison of I_c, n, RRR, and Q_{hyst} are presented. The results from the two laboratories are in good agreement. The random difference is attributed to the variation along the sampling-wire length. This cross-checking of large number of samples provides a set of interesting and reassuring data, which confirms statistically significant agreement between the two laboratories.

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Authors' biographies not available at the time of publication.