

# Repeatability of Critical-Current Measurements on Nb<sub>3</sub>Sn and Nb-Ti Wires

L. F. Goodrich, L. T. Medina, and T. C. Stauffer

National Institute of Standards and Technology (NIST), Boulder, CO USA

**Abstract**— A varying degree of repeatability has been observed in critical-current ( $I_c$ ) measurements of Nb<sub>3</sub>Sn and Nb-Ti wires as a function of the number of thermal cycles from room temperature to 4 K. The increase of  $I_c$  between the first and second thermal cycle can be 1% to 2% at 12 T for Nb<sub>3</sub>Sn wires. This was observed on a Nb<sub>3</sub>Sn wire by all four laboratories that participated in a recent interlaboratory comparison conducted in the International Thermonuclear Experimental Reactor (ITER) project. These data indicate that if  $I_c$  changes beyond the error limits, it increases fairly monotonically with thermal cycling until it eventually saturates. In contrast, the  $I_c$  of a Nb-Ti wire is very repeatable with thermal cycling. This suggests that the effect on the Nb<sub>3</sub>Sn wire is due to its strain sensitivity. Most of these data were taken with the sample on a Ti-6Al-4V measurement mandrel. This study also investigated the repeatability of  $I_c$  measurements using other mandrel materials. The increase in  $I_c$  of Nb<sub>3</sub>Sn wire could enhance the performance of some applications. However, the lack of repeatability in  $I_c$  measurements on Nb<sub>3</sub>Sn wires is a limitation in precise interlaboratory comparisons.

## I. INTRODUCTION

Multiple critical-current ( $I_c$ ) measurements on different samples have produced some results which indicate a degree of training with each thermal cycle imposed on a given specimen. The type of training observed here is a systematic change in  $I_c$  of a given specimen with each thermal cycle, while  $I_c$  is highly repeatable within each thermal cycle. The different samples used in this experiment include internal-tin processed Nb<sub>3</sub>Sn (Y), bronze processed Nb<sub>3</sub>Sn (Z), and the NIST Nb-Ti (S) Standard Reference Material (SRM-1457). At least four specimens of each sample were mounted on various measurement-mandrel materials for  $I_c$  testing. Measurement mandrels used in this training experiment were: Ti-6Al-4V (% by mass, Ti-6-4), a fiberglass epoxy composite (G-10), stainless steel 316L, and Incoloy 908. All of the different measurement mandrels were constructed to provide the same coil sample geometry. The direction of the specimen current was selected so that the Lorentz force was into the measurement mandrel. All of the samples were mounted on Ti-6-4 measurement mandrels and some were mounted on additional materials. Only the Nb-Ti wire was mounted on the stainless steel and Incoloy mandrels.

As a result of a number of interlaboratory comparisons, the International Thermonuclear Experimental Reactor (ITER) project has adopted a standard  $I_c$  measurement procedure [1] that uses a Ti-6-4 reaction and measurement mandrel. This standard reduced the spread of data by about a factor of 10, from 30% to 3.3% [2]. With this improved accuracy, the subtle increase of

thermal-cycle training can be observed and can be a limiting factor in precise interlaboratory comparisons. All four international laboratories that participated in the recent ITER interlaboratory comparison observed similar increases of about 2% in  $I_c$  of Nb<sub>3</sub>Sn wires with a subsequent thermal cycle. We had observed thermal-cycle training and postulated a mechanism for this effect [1] prior to the international interlaboratory comparison and thus were able to inform the ITER participants of the need to keep track of the thermal cycles of each specimen.

In this paper, we investigated the effects of a number of parameters on training. The differential thermal contraction between the specimen and the measurement mandrel will change the strain state of the specimen. This differential contraction is expected to be about 0.12% ± 0.03% for specimens on Ti-6-4 and about 0.00% ± 0.03% for specimens on thin-walled G-10 tubes [3]. The dynamic differential contraction between these two materials may be more than 0.12% during the cooling or warming cycles due to transient temperature differences between the mandrel and wire. The specimen undergoes hoop stress and elongation as it is cooled to 4 K when constrained on a Ti-6-4 mandrel and the subsequent thermal cycles amount to stress-strain fatigue cycles to the same 4-K strain point. We suspect that these fatigue cycles have a cumulative effect on the total strain state, transverse and axial, of the Nb<sub>3</sub>Sn filaments. A relief of some of the strain would result in an increased  $I_c$  with each thermal cycle until it reaches a saturated value. The copper component of the wire (and maybe also the bronze) will be very soft and it will yield during the first few thermal cycles. The result of any yielding will be that the initial loading of the wire will occur at progressively lower temperatures through these cycles. Coupled with the strong temperature dependence of the elastic modulus of Nb<sub>3</sub>Sn, associated with a martensitic phase transformation [4], this may result in mechanical hysteresis and a change in the total strain state of the Nb<sub>3</sub>Sn between thermal cycles. The complex thermal-mechanical nature of this system makes a detailed analysis beyond scope of this paper. Further evidence that this mechanism is correct would be obtained by observing the lack of thermal cycle training in Nb<sub>3</sub>Sn wires measured on G-10 mandrels and in Nb-Ti wires measured on Ti-6-4 and G-10 mandrels. This is the focus of this paper.

## II. EXPERIMENTAL

$I_c$  measurements were made on three pairs of voltage taps, each separated by 25 cm. In most cases, we used the average  $I_c$  of these three pairs. For a few specimens, 50 or 75 cm tap separations were used to be consistent in interlaboratory comparisons. For the Nb<sub>3</sub>Sn specimens, we made  $I_c$  measurements at magnetic fields of 10, 11, and 12 T, temperatures of about 4.0 and 4.2 K, and a criterion of 10 μV/m.

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Typically, three determinations of  $I_c$  were made at each field and temperature. For the Nb-Ti specimens, similar measurements were made, but at magnetic fields of 4, 6, and 8 T. The temperature dependence of  $I_c$  was assumed to be linear at each field, and the six determinations were fit to determine a tap average, a single value for each tap at 4.2 K. For specimens with three pairs of 25 cm taps, the specimen average was calculated by averaging the three tap averages. For specimens with a 50 or 75 cm tap pair, the specimen average was set to the tap average of the longest pair. The primary data for this paper will be the 8 T data for Nb-Ti and 12 T for Nb<sub>3</sub>Sn wires. The estimated uncertainty of these  $I_c$  measurements is  $\pm 2\%$  for Nb<sub>3</sub>Sn wires and  $\pm 1\%$  for Nb-Ti wires. The estimated precision is  $\pm 0.1\%$ .

One important feature of the standard  $I_c$  measurement procedure employed here is the modular design of the specimen mandrel and its standard mount to the test fixture [1]. This allows the specimen to be removed from the test fixture without detaching the current or voltage connections from the specimen. Some of the specimens were unmounted and remounted to the test fixture between thermal cycles and others were not. This did not appear to affect the repeatability of  $I_c$ . Specimens were always warmed to room temperature between each thermal cycle.

### III. RESULTS

#### A. Nb<sub>3</sub>Sn

In investigating the Nb<sub>3</sub>Sn wires, we observed training of the specimen when measured on Ti-6-4. All the Nb<sub>3</sub>Sn specimens mounted on Ti-6-4, with or without epoxy bonding, trained by more than 2% after 3 to 6 thermal cycles. The data presented in Fig. 1 are for the bronze processed (Z) Nb<sub>3</sub>Sn wires at 12 T. The differences in the specimen average  $I_c$ s are relative to their initial values. The specimens shown in Fig. 1, two with and two without bonding, all trained in a similar manner. The training effect shown in Fig. 1 may saturate at a 4% total increase in  $I_c$  at 12 T. In general, the change in  $I_c$  was less at 10 T. For example, the effect on specimen Z1 was 3.5% at 10 T and 4.2% at 12 T on the sixth thermal cycle. This suggests that the source of this training is strain; thus the change in  $I_c$  will likely be more at magnetic fields higher than 12 T [5].

Fig. 2 shows the data for the internal-tin processed (Y) Nb<sub>3</sub>Sn wires at 12 T. The four specimens on Ti-6-4 shown here are relatively consistent with the results from above. Specimen Y4 may not have been completely tensioned on the mandrel and the resulting slack accommodated some of the differential contraction which reduced the change in  $I_c$ . The other two Y specimens were epoxied to and measured on G-10 mandrels which showed a cumulative change of less than 1% for six thermal cycles. When a specimen is epoxied to the mandrel, there is no slack. These results indicate that differential thermal contraction may be necessary to create this type of training.

Within a given thermal cycle, the  $I_c$  of a Nb<sub>3</sub>Sn wire mounted on Ti-6-4 is very repeatable. Fig. 3 shows the difference of individual  $I_c$  measurements from their average values on specimen Z4 for the fourth thermal cycle as a function of

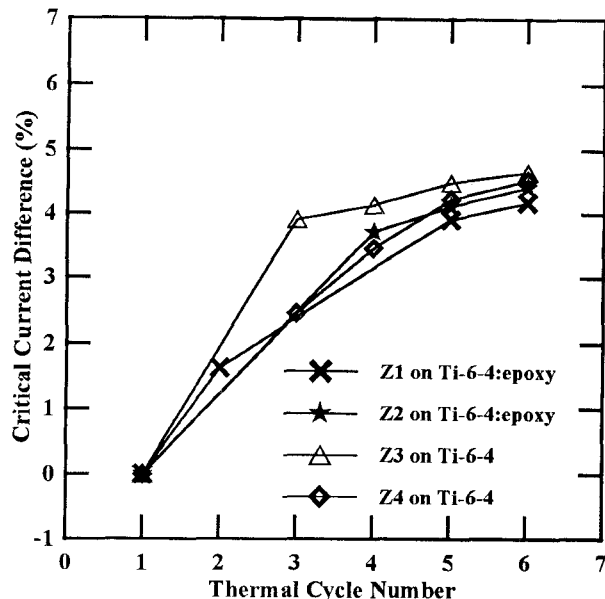


Fig. 1. Percent difference (from initial value) in the specimen average  $I_c$  of the bronze processed Nb<sub>3</sub>Sn specimens at 12 T versus the thermal cycle number.

determination at 12 T. There is no indication of an increase in  $I_c$  with each determination in the sequence that would account for the observed 0.75% increase in the next thermal cycle.

#### B. Nb-Ti

The cumulative effect of thermal cycling is less than 0.3% for Nb-Ti wires (S) measured four to six times on Ti-6-4 mandrels and multiple times on G-10 mandrels. This Nb-Ti wire is expected to be representative of all others in this respect. Fig. 4 presents the data for Nb-Ti wires at 8 T. The change in  $I_c$  is less

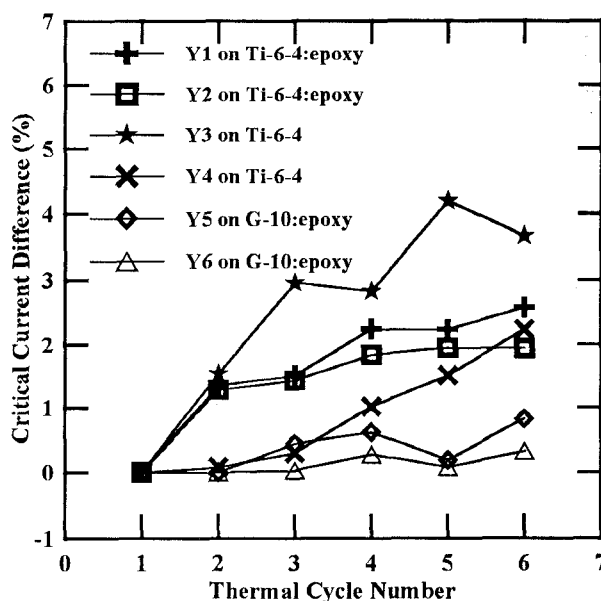


Fig. 2. Percent difference (from initial value) in the specimen average  $I_c$  of the internal-tin processed Nb<sub>3</sub>Sn specimens at 12 T versus the thermal cycle number.

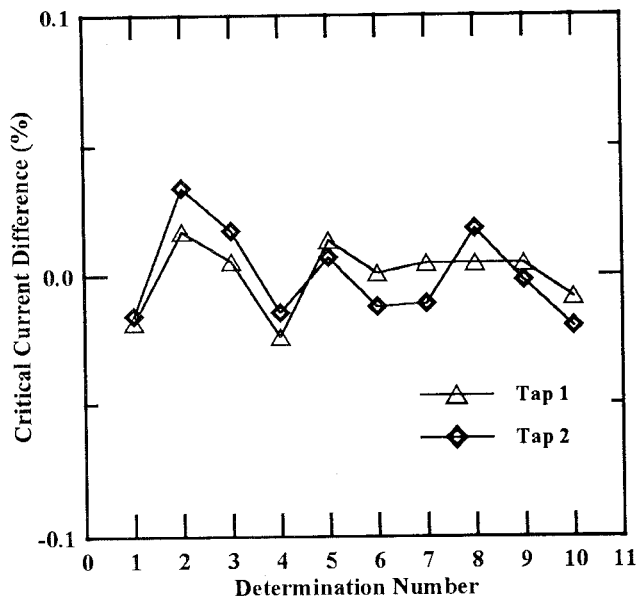


Fig. 3. Percent difference (from their average values) in the individual  $I_c$  determinations on each of two voltage tap pairs during the fourth thermal cycle of  $Nb_3Sn$  specimen Z4 at 12 T.

than 0.3% for all Nb-Ti specimens regardless of mandrel material and bonding. The measurements of each of these four Nb-Ti specimens were spread over a period of 1 to 2 years and were made on two test fixtures, so some of this variation may be due to small changes in the measurement system calibration. These results indicate that Nb-Ti wires do not show this type of training and that the measurement system is capable of giving consistent  $I_c$ s with thermal cycles and remounting of specimens. Since Nb-Ti wires are relatively strain insensitive compared to  $Nb_3Sn$  wires, the remounting result on Nb-Ti wires does not carry over to  $Nb_3Sn$  wires.

Another aspect of this experiment was testing one Nb-Ti specimen on different mandrel materials with the same coil sample geometry. Fig. 5 shows the percent difference in  $I_c$  versus the measurement mandrel material. S4 was measured multiple times on G-10, Incoloy 908, Ti-6-4, and stainless steel 316L. There is a fairly significant change in the measured  $I_c$ , which was 14.4% lower (off scale) at 8 T, apparently due to the magnetic properties of the Incoloy mandrel. This is equivalent to a 0.38 T increase in the applied magnetic field. The reductions of  $I_c$  at magnetic fields of 2, 4, and 6 T (equivalent fields of 0.42, 0.39, and 0.38 T respectively) were also very consistent with a 0.38 T increase in the magnetic field experienced by the specimen at 8 T. Considering the continuity of the flux density  $B$ , this suggests that the additional magnetic field from the saturated magnetization of the Incoloy has this effective value in the groove where the specimen resides. Thus, it is not advisable to consider using Incoloy as a measurement mandrel material else the  $I_c$  measurements of Nb-Ti and  $Nb_3Sn$  wires will be biased. There was a relatively minor magnetic enhancement when using the stainless steel mandrel. An additional field of about 0.02 T would account for the observed 0.8% decrease in the measured  $I_c$  on the stainless steel mandrel.

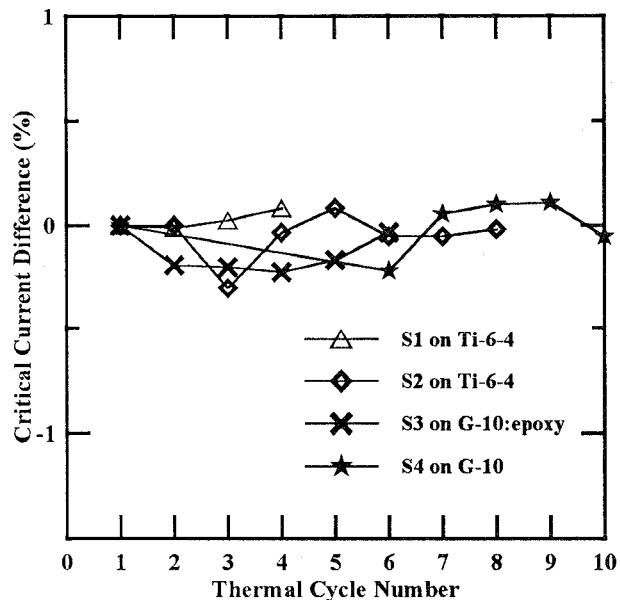


Fig. 4. Percent difference (from initial value) in the specimen average  $I_c$  of the Nb-Ti specimens at 8 T versus the thermal cycle number.

Making  $I_c$  measurements of a  $Nb_3Sn$  specimen on various measurement mandrel materials would also introduce changes in  $I_c$  due to the differential contraction between the mandrel and the specimen, which puts the wire in different strain states on different mandrels. For example, two  $Nb_3Sn$  specimens were measured on Ti-6-4 mandrels and then transferred to G-10 mandrels where they were measured again. On average, the  $I_c$  was 9.8% lower at 12 T on the G-10 mandrel. In contrast, the change in  $I_c$  of a Nb-Ti specimen was less than 0.3% for measurements on these two mandrel materials.

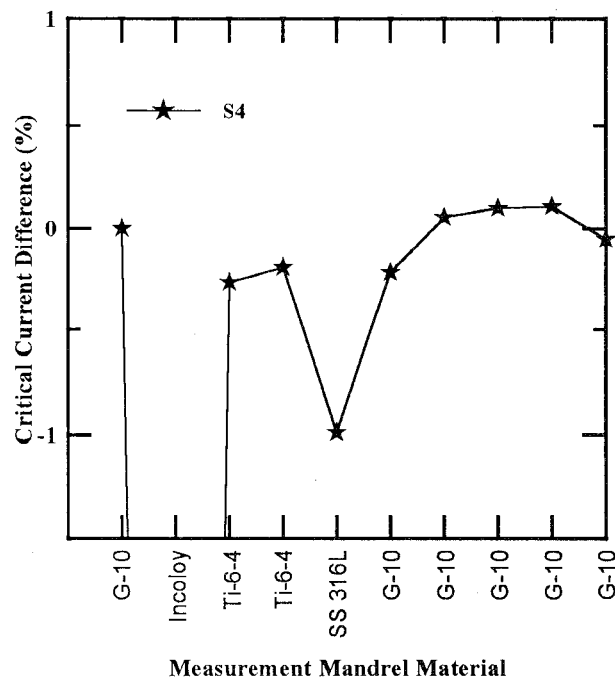


Fig. 5. Percent difference (from initial value) in the specimen average  $I_c$  of Nb-Ti specimen S4 at 8 T versus the sequence of measurement mandrel materials.

### C. Complementary effects within a specimen

A complementary effect was observed among the  $I_c$ s of the three pairs of voltage taps on a given specimen in cases where the specimen was not bonded with epoxy to the measurement mandrel. In other words,  $I_c$  in one region can go up at the expense of it going down in another region. Fig. 6 shows the change in  $I_c$  for each tap pair relative to the initial specimen average as a function of thermal cycle. Between thermal cycles 1 and 2, the  $I_c$  of tap 3 went down and the  $I_c$  of tap 2 went up. This could be explained by a redistribution of wire tension along a specimen. The redistribution of wire tension outside the instrumented regions of the specimen may also lead to non-monotonic and non-saturating observations.

In contrast, when a specimen is bonded to the mandrel, the  $I_c$ s of the various regions of the specimen track each other through the thermal cycles. As shown in Fig. 7, the relationship between the  $I_c$ s of the two 25 cm voltage tap pairs on this bonded specimen remains virtually the same through the thermal cycles because the tension cannot be redistributed. These results were consistent with observations on other specimens.

### V. CONCLUSIONS

Internal-tin and bronze processed  $Nb_3Sn$  wires exhibit thermal-cycle training of the  $I_c$  when mounted on a Ti-6Al-4V measurement mandrel material whether or not the wires were bonded to the mandrel with epoxy. The increase between the first and second thermal cycle can be 1% to 2% at 12 T. This effect seems to saturate after a total increase in  $I_c$  of 2% to 5%. This is a relatively small effect, but it can be a limiting factor in precise interlaboratory comparisons. However, the increase in  $I_c$  of  $Nb_3Sn$  wires could enhance the performance of some applications.  $Nb_3Sn$  wires mounted on fiberglass-epoxy

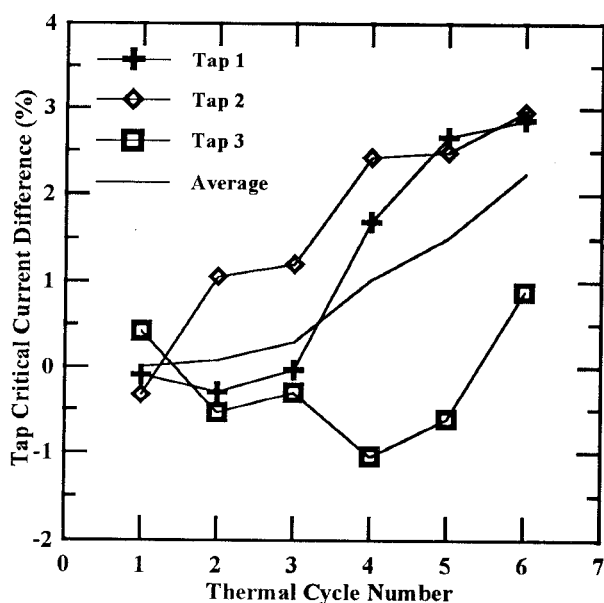


Fig. 6. Percent difference (from the initial specimen average) in the tap average  $I_c$  of Y4 which was not bonded to the Ti-6-4 mandrel.

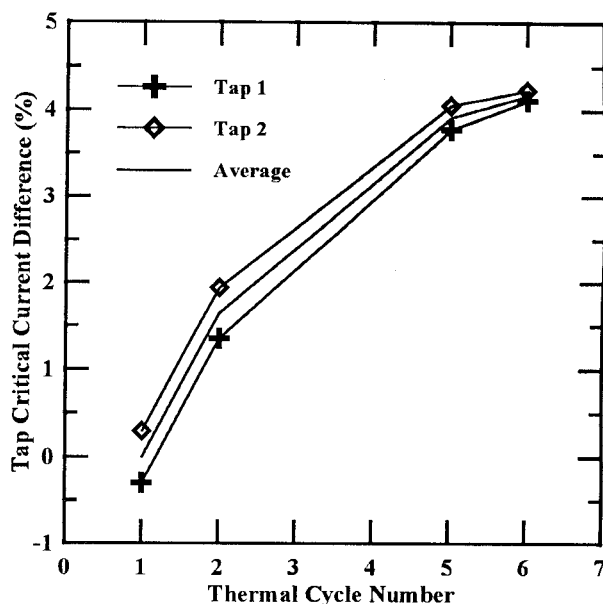


Fig. 7. Percent difference (from the initial specimen average) in the tap average  $I_c$  of Z1 which was bonded to the Ti-6-4 mandrel.

composites (G-10) did not exhibit training; the change in  $I_c$  was less than 1% after six thermal cycles. One advantage of Ti-6-4 over G-10 is that the same mandrel can be used for reaction and measurement, which avoids possible damage during sample transfer, that may outweigh the disadvantage of thermal-cycle training in routine measurements. The  $I_c$  of a Nb-Ti wire changed by less than 0.3% through numerous thermal cycles when mounted on Ti-6-4 and thin-walled fiberglass epoxy composite measurement mandrels. These observations are consistent with the thermal-mechanical hysteresis mechanism proposed here.

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