First VAMAS USA Interlaboratory Comparison of High Temperature Superconductor Critical Current Measurements

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Abstract-**We conducted an interlaboratory comparison of critical current (I_c) measurements on $Bi_2Sr_2Ca_2Cu_3O_{10}$ tapes (2223). This study includes measurements from six participating US laboratories, with NIST as the central, organizing laboratory. A number of specimens were prepared with different degrees of instrumentation to isolate sources of variability. Most of the specimens were pre-measured by NIST to reduce uncertainties due to sample variability. Different specimen routing patterns among the laboratories were implemented to isolate sources of variability due to the specimen's measurement history. This study is similar to other VAMAS intercomparisons being performed in Japan and Europe and is the first internationally cooperative interlaboratory comparison of HTS (High Temperature Superconductors) I_c measurements. These are the first steps towards developing standard measurement procedures for HTS.**

I. Introduction

In this paper we will give an outline of the entire interlaboratory comparison and include the results we have obtained thus far in context with the entire comparison. The background research for this intercomparison can be found in [1]. Similar comparisons are being conducted independently in Japan and Europe [2,3] under the Versailles Project on Advanced Materials and Standards (VAMAS). Although parts of this comparison are incomplete, we have sufficient data to present results indicate the homogeneity of the critical current (I_c) within a sample, the significance of various source of variability, and the level of agreement among the laboratories under various conditions.

We obtained two samples from different manufacturers, which we call Sample X and Sample Y, of Ag-sheathed $Bi_2Sr_2Ca_2Cu_3O_{10}$ tapes (2223). Each sample consisted of 60 specimens which were each 42 mm long. Further details on the materials are not essential for this paper and are omitted by agreement with the manufacturers. These 60 specimens were separated into six different classes, A-F, in an attempt to isolate sources of variability in High Temperature Superconductors (HTS) I_c measurements (see Table I). Four parameters were used to separate the specimens into these classes. These parameters were the routing of the specimens through the laboratories and whether the specimens had been pre-measured, pre-mounted, or pre-instrumented. There were two different ways that we routed specimens in this comparison.

Manuscript received October 17, 1994.

TABLE I
SPECIMEN CLASS DEFINITIONS

Class	Pre-measured	Pre-mounted (substrate material)	Pre-instrumented	Routing
A	No	No	No	Parallel
B	Yes	No	Partial [a]	Parallel
C	Yes	G-10	Yes	Parallel
D	Yes	G-10	Yes	Series
E	Yes	Brass	Yes	Series
F	Yes	Brass	Yes	Parallel

[a] These specimens had pressure current contacts, soldered voltage taps, and were measured on G-10 but were not bonded to anything

Some specimens were *series routed*, meaning that they were sent from NIST, measured at each laboratory in sequence, and then returned to NIST. Other specimens were *parallel routed*, meaning that they were sent from NIST, measured by a participating laboratory, and then returned to NIST. In the parallel routing, each laboratory measured different specimens, and no specimen was sent to more than one laboratory other than NIST. *Pre-measured* means that the specimen was measured by NIST before being sent out to any of the laboratories. *Pre-mounted* means that NIST bonded a specimen to a substrate material. *Pre-instrumented* means that the voltage (1.5 cm tap separation) and current leads have been soldered to the specimen. Table I shows the detailed definitions of the specimen classes.

The experiment was divided into two stages: a preliminary stage and a main stage. The preliminary stage consisted of four parallel routed specimens for each lab; one Class C and one Class F specimen from each of the two samples (X and Y). Each lab measured the four specimens at temperatures of 4.2 and 77 K with zero magnetic field.

The main stage of the experiment had both series and parallel routed specimens. One specimen from each of Class A, D, and E from each sample was measured in zero magnetic field at temperatures of 4.2 and 77 K. One specimen from each of Class B, C, D, and E from each sample was measured as a function of magnetic field at temperatures of 4.2 K and 77 K. The field measurements were done from 0 to 8 T at 4.2 K and 0 to 1 T at 77 K. Each laboratory, excluding NIST, measured a total of 18 specimens (10 in zero magnetic field only). We chose to have a large number of specimens and the complex class and routing system in order to obtain sufficient data to ensure statistically meaningful results.

II. Sources Of Variability

For the purpose of this discussion we have separated the sources of variability in critical current measurements into four groups: sample, mounting, measurement, and damage. Sample variability includes: sample inhomogeneity, repeatability, training effects, and hysteresis of magnetic field, current, and temperature. Mounting variability includes: solder temperature, bonding agent, and substrate material. Measurement variability includes: technique, and precision and accuracy of voltage, current, magnetic field, temperature, and tap separation. Damage variability includes: thermal cycling, time, handling, and shipping. These are partial lists of possible parameters in each group.

After the data were compiled we identified three sources of severe damage variability:

1. Bubbles in the Ag sheath between or near the voltage taps. These bubbles were probably due to rapid warming of the liquid cryogen which had seeped into the specimen resulting in a gas that expanded faster than it could escape.

2. Specimens coming loose from the substrate. This occurred only on the brass substrates and could be due to poor bonding or differential thermal contraction. We prepared the specimen for bonding to the brass substrate by roughing the substrate surface with sandpaper and degreasing it.

3. Specimen damage, possibly due to the specimen hitting the dewar.

Each of these sources of severe damage variability caused at least a 10% reduction of critical current from the initial NIST measurement. We can distinguish these severe sources of damage variability from the other sources of variability by correlating visible specimen anomalies with reductions in critical current. There are ways to reduce the severe sources of damage variability listed above: preliminary thermal cycling to identify and subsequently eliminate specimens that are prone to bubble, eliminating brass as a substrate material with epoxy as the bonding agent, and protecting the specimens during measurements so they cannot inadvertently hit the dewar. Data taken on damaged specimens are treated as outliers in the analysis presented below and thus are removed from the statistics.

III. Results

Fig. 1 shows a histogram of NIST's initial measured critical current from the preliminary and main stages of the experiment for specimens from Sample X at a temperature of 77 K with zero magnetic field. This histogram shows a remarkably narrow distribution of less than a 9% range in the measured critical current for 24 specimens and a coefficient of variation (standard deviation divided by the average) of 2.5%. These 24 specimens were from Classes B, C, D, E, and F.

Table II gives the statistics for NIST's initial critical current measurements on the X and Y specimens at 4.2 and 77 K with zero magnetic field. These 24 specimens were from Classes B, C, D, E, and F. The outlying data from a specimen from

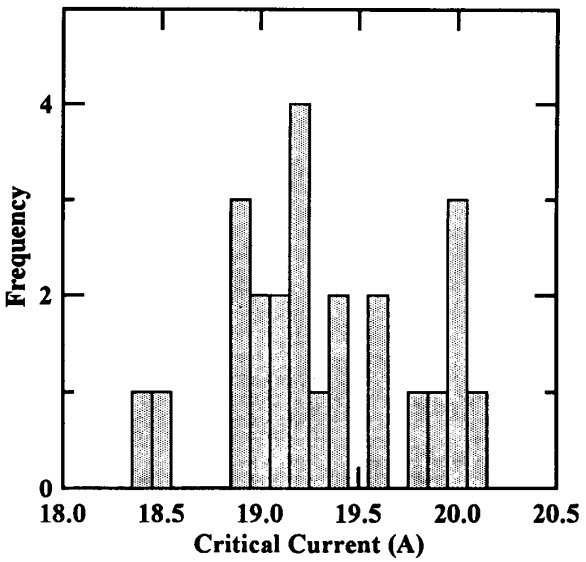

Fig. 1. Distribution of NIST's initial critical current measurements from the preliminary and main stages of the experiment.

TABLE II
Summary Statistics Based on Initial NIST Measurements for Samples X and Y

	Liquid Nitrogen		Liquid Helium	
I_c at 1 μV/cm	Sample X	Sample Y	Sample X	Sample Y
Min, A	18.36	12.14	88.50	79.62
Max, A	20.02	16.94	95.11	106.1
Range, %	8.63	34.42	7.24	29.36
Standard Dev., A	0.48	1.20	1.80	6.33
Average, A	19.28	13.95	91.37	90.12
Coef. of Var, %	2.46	8.60	1.97	7.03
# of Points	24	23	24	23
n-value Average	29.6	28.5	36.2	28.9

Sample Y were removed from the statistics. We did not observe a systematic trend with different mounting substrate materials and different degrees of instrumentation. Therefore we think the distributions are an indication of sample variability. The estimated total uncertainty of the NIST I_c measurements on HTS samples is ±1%.

Fig. 2 shows the normalized critical current at 1 μV/cm, 77 K, and zero magnetic field as a function of the measurement laboratory for specimens from Samples X and Y. These specimens were in the preliminary stage of the experiment and were parallel routed (Class C and F). The normalization was done with respect to the NIST initial measurement. The plot indicates that most specimens did not degrade by more than 4%, while some specimens significantly degraded by the time they returned to NIST for remeasurement. All specimens that degraded to less than 95% of the original NIST measurement had a visible specimen anomaly.

Fig. 3 is the same as Fig. 2, except with an expanded Y axis to illustrate the agreement between the measurements on specimens that were not damaged. The plot indicates that the

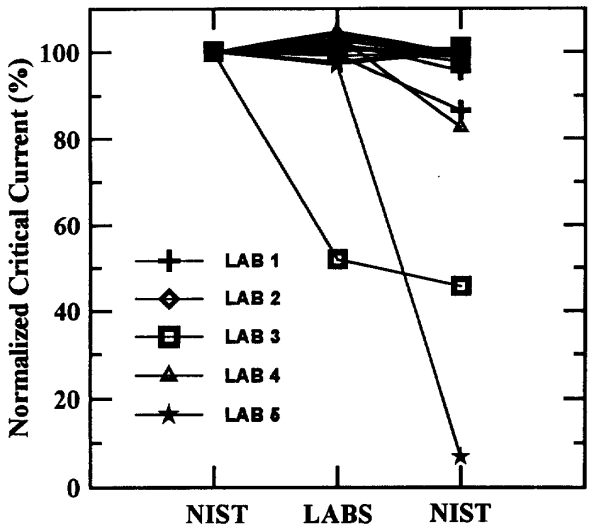

Fig. 2. Normalized I_c at 1 μV/cm, 77 K, and zero magnetic field versus measurement laboratory for specimens from Samples X and Y.

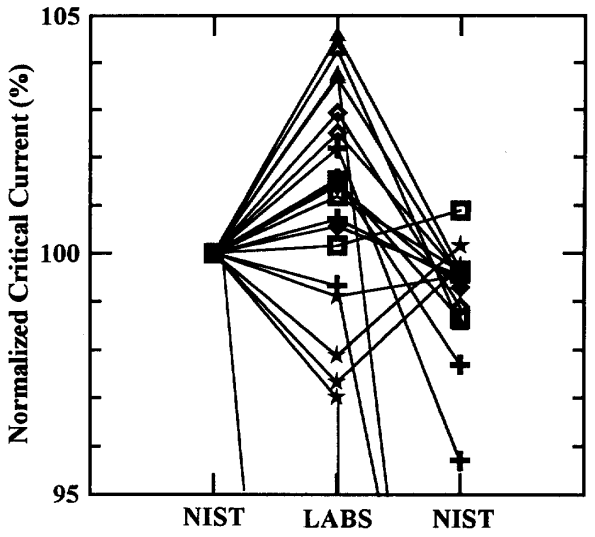

Fig. 3. Normalized I_c at 1 μV/cm, 77 K, and zero magnetic field versus measurement laboratory for specimens from Samples X and Y on an expanded scale.

distribution of the participating laboratories' measurements was within $\pm 5\%$ of the initial NIST measurement. The variability of the laboratory measurements is larger than the final NIST measurements due to the differences among the measurement systems. This part of the experiment was designed to focus on the bias and variability of individual laboratories' measurements. The time between the initial and final measurements was between 3.5 and 11 weeks on specimens from different laboratories. This indicates that specimen degradation due to time may be relatively insignificant. Similar results to those presented in Fig. 3 were obtained on parallel routed specimens in the main stage versus magnetic field; however there was an increase in variability.

Table III gives summary statistics for the normalized critical current at 1 μV/cm at temperatures of 4.2 and 77 K for specimens used in the preliminary stage of the experiment. The table contains statistics from the participating laboratories as well as the NIST re-measurement. The result of the preliminary stage showed good agreement among the laboratories with Class C and F specimens in which all of the sources of variability were minimized.

Table IV contains summary statistics for the Class A and B specimens in the main stage. These specimens were parallel routed, with no pre-mounted substrate material. This part of the comparison focuses on the increased variability due to laboratory measurement and mounting.

The Sample Y measurements at liquid helium temperature contained an outlier (a damaged specimen) which was removed from the statistics. The main stage of this comparison is not yet completed; therefore the results may lack statistical significance. Comparing the partial results in Table IV with the results in Table II indicate that the variability of the laboratory determined I_c of Class A and B specimens may be 1.9 times that of the sample variability. Unless these statistics are misleading, this indicates that there is a significant contribution from laboratory measurement and mounting variability.

Fig. 4 shows the series routed (Class D and E) normalized critical current as a function of the measurement laboratory sequence at zero field and a temperature of 77 K. This part of the comparison focuses on damage variability. The lowest two points are outliers. The results, though incomplete, indicate the continuation of the trends observed in the parallel routed specimens on Figs. 2 and 3, a slight decrease in I_c and some outliers.

TABLE III
SUMMARY STATISTICS FOR PRELIMINARY STAGE
(CLASS C AND F, SAMPLES X AND Y)

Normalized	Liquid Nitrogen		Liquid Helium	
I_c at	LABS	NIST	LABS	NIST
1 μV/cm	(5)	re-measure	(5)	re-measure
# of Points	19	16	19	16
Average, %	101.1	99.1	98.3	98.9
Range, %	7.53	5.19	10.7	3.30
Coef. of Var, %	2.26	1.16	3.03	1.09

TABLE IV
SUMMARY STATISTICS FOR LABORATORY I_c DATA ON
CLASS A AND B SPECIMENS

	Liquid Nitrogen		Liquid Helium	
I_c at 1 μV/cm	Sample X	Sample Y	Sample X	Sample Y
Min, A	17.5	11.40	87.58	76.3
Max, A	21.01	16.29	96.44	99.47
Range, %	18.62	4.89	8.86	23.17
Standard Dev., A	1.23	1.60	3.53	8.96
Average, A	18.85	12.98	90.36	84.03
Coef. of Var, %	6.55	12.29	3.91	10.66
# of Points	7	7	7	6

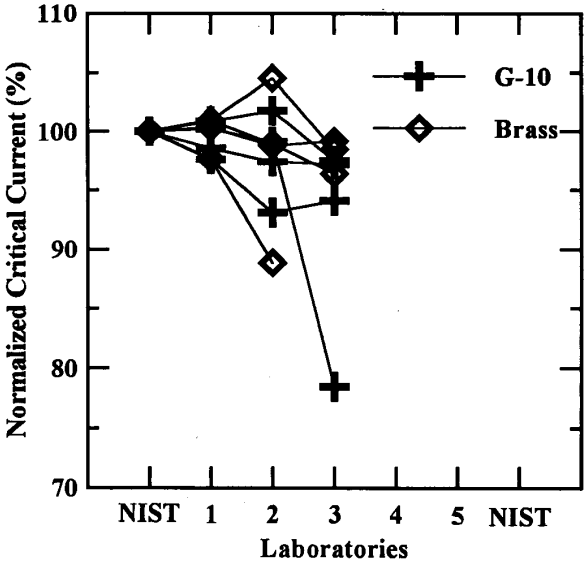

Fig. 4. Critical current from series routed specimens (Class D and E) as a function of measurement laboratory sequence at zero field and 77 K.

IV. Discussion

This interlaboratory comparison of critical current measurements on HTS superconductors delivered a considerable amount of quantitative and qualitative data. There are many ways to analyze these data and further analysis will be done when this comparison is completed. Our experience during this experiment suggests that the measurement facilities of some laboratories are overloaded or some of the measurements that we requested are not routine. We designed the preliminary stage of the experiment to give each participating laboratory a chance to make modifications to their specimen probes and make measurements in parallel before they received the tightly scheduled series specimens. However, the schedule was not maintained.

V. Conclusions

We measured specimens mounted in different ways (solder contacts, pressure contacts) using different substrates (none, G-10 (fiberglass-reinforced epoxy), or brass) and obtained consistent results within what was expected to be sample variability. The preliminary stage had the most ideal conditions because all of the specimens were pre-mounted, pre-instrumented, and parallel routed, which minimized all of the sources of variability. The observed coefficient of variation of I_c values in this stage was about 2.3% at 77 K and 3.0% at 4.2 K in zero magnetic field. This would be adequate for I_c measurements on HTS; however some outlying data were removed from these statistics, and this part of the comparison was conducted under the most ideal conditions. To reduce outlier data in the future, we recommend the following: not using specimens bonded to brass substrates with epoxy, protecting specimens from damage on the probe, and thermally cycling specimens to remove those prone to bubbling. The measurement and mounting variability, which was studied on specimens mounted by each laboratory, may cause I_c variation that is much larger than the numbers above. The results of the series routed specimens, which focuses on damage variability, though incomplete, indicate that some specimens can retain more than 94% of their I_c through measurements at four laboratories at 4 and 77 K.

Acknowledgment

The authors thank American Superconductor Corp. and Intermagnetics General Corp. for generously providing us with samples for this interlaboratory comparison. The six laboratories that participated in this comparison were: American Superconductor Corporation, Florida State University, Intermagnetics General Corporation, National Institute of Standards and Technology, Oxford Superconducting Technology, and the University of Wisconsin. All of the participating laboratories gave of their time, expertise, and facilities to this important effort. Neither NIST nor the participating laboratories obtained external funding specifically for this work. Certain commercial products mentioned in this paper might be indirectly identified by their particular properties. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the products are necessarily the best available for the purpose.

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