CRITICAL CURRENT MEASUREMENTS ON A NbTi SUPERCONDUCTING WIRE STANDARD REFERENCE MATERIAL*

L. F. Goodrich, D. F. Vecchia, E. S. Pittman, and A. F. Clark

Electromagnetic Technology and Statistical Engineering Divisions
National Bureau of Standards
Boulder, Colorado

INTRODUCTION

The National Bureau of Standards is producing a standard reference material (SRM) for the measurement of the superconducting critical current (I_c). This SRM and the recently adopted American Society for Testing and Materials (ASTM) standard test method (B714-82) will aid both the commerce and technology of superconductors through the promotion of more uniform measurements. The SRM will serve as an artifact for interlaboratory comparison to further advance the consensus and evolution of the new test method. The general use and philosophy of an SRM are given in Ref. 1.

To perform well as an SRM, the conductor chosen should be as homogeneous as possible. Conductors were purchased from the inventories of each of the United States wire manufacturers. Each conductor was selected by the manufacturer as a good candidate for an SRM. Preliminary screening measurements were performed on each conductor to determine the short- and long-range spatial variations in the critical current. The choice of the SRM was based primarily on these data.

The conductor selected for the critical current SRM was despooled onto 500 distribution spools, each with ~2.2 m of wire.

*Work supported in part by the NBS Office of Standard Reference Materials and the Department of Energy.

Adv. Cryog. Eng. - Materials, Vol. 30,

Plenum Press, New York (1984), 953-960.

Measurements on a sample of the spools were used to determine the likely outcomes if the spools for distribution were measured. The statistical analysis presented is for data taken at 4.07 K. A complete description of the measurement procedure, temperature dependence, and statistical analysis will be reported elsewhere.[2] This analysis will be based on the user making one measurement, on one spool, at one of the given magnetic fields (2, 4, 6, and 8 T) and at any temperature from 3.90 to 4.24 K.

EXPERIMENT

The only significant change from the common instrumentation[3] used to measure I_c, was the addition of a digital processing oscilloscope. This allowed the automation of the data analysis and higher precision in the processing of the voltage-current (V-I) curves than using an analog X-Y recorder alone. A number of acquisition and analysis variables were identified and their effect on the precision and accuracy of the critical current measurement determined. The acquisition variables considered were: current ramp rate, digital sampling rate, and voltage filtering and amplification. The analysis variables and corrections considered were: corrections for the inductive voltage and changes in thermoelectric voltage, and the number of points in the curve fitting. A correction for the magnetic field profile was made to I_c measured on adjacent voltage taps. The hydrostatic head and stratification of the liquid helium bath were also measured and the correct liquid helium vapor pressure used for the temperature determination.

The principal sample geometry used was a helical coil. There are many tradeoffs for each geometry (e.g., long straight, short straight, hairpin, etc.), but the coil seemed the best for this type of testing. With the coil, a number of segments of a given specimen could be measured to test the short range variations. Also the problems of negative voltage,[4] magnetic field angle and field uniformity are less in this geometry. The only major problem is the effect of bending strain which is not well known and is hard to measure[2] for NbTi superconductors; however, an upper limit can be put on the effect using uniaxial strain data.[5] The plastic flow of the copper matrix,[6] which would reduce the bending strain on the NbTi filaments, would make the estimated correction based on uniaxial strain data an upper limit. With a coil diameter of 3.2 cm and a sample of 0.05 cm diameter, the peak bending strain would be approximately 1.6%, assuming the neutral axis does not shift. A uniaxial strain of 1.6% would decrease I_c by about 8% at a magnetic field of 8 T. The integral of this effect over the cross section results in a 2% decrease in the overall value of I_c of the wire assuming the twist pitch is longer than the current transfer length,[6] which is a reasonable assumption for these NbTi superconductors. Because the bending strain correction is not exactly known, the correction of the I_c data to the unbent state was not made, but it should be less than 2% at 8 T and even less at the lower magnetic fields.

The calibration of the instruments used to measure voltage, current, magnetic field, pressure, and length was performed.[2] The estimated systematic errors (inaccuracies) and random errors (precision limits) are given in Table 1. The random errors are included in the observed variation in the critical current and for this reason were separated from the systematic errors. The error in each of the critical current variables is expressed in terms of the resultant percentage error in the critical current at each of the magnetic fields. These errors were estimated using the known dependence of the critical current on each of the variables. The periodic and random deviation (PARD) of the current and magnetic field are not included because the sample current source was a battery power supply and the magnet was used in persistent mode. The sum and the root-mean-square of the estimated errors are given at the bottom of the table for each magnetic field.

PRELIMINARY SCREENING

The selection, from the five candidate conductors for the SRM, was based on the best balance of properties for use as an SRM. The chosen conductor may not be the best for any other

Table 1. Systematic and Random Errors Expressed in Percent Error in I_c at Magnetic Fields of 2, 4, 6, and 8 T.

Variable	Systematic error (%) 2 T	4 T	6 T	8 T	Random error (%) 2 T	4 T	6 T	8 T
Current	.25	.25	.25	.25	.05	.05	.05	.05
Electric field	.05	.05	.05	.07	.05	.05	.05	.07
Magnetic field	.10	.12	.21	.45	.04	.05	.05	.09
Temperature	.02	.02	.03	.04	.06	.08	.10	.17
Magnetic field profile	.02	.03	.05	.10				
Magnetic field angle	.10	.10	.10	.10				
Tensile strain	.15	.15	.15	.15	.07	.07	.07	.07
$\Sigma\Delta$	.69	.72	.84	1.16	.27	.30	.32	.45
$\sqrt{\Sigma(\Delta^2)}$	.33	.34	.38	.56	.12	.14	.15	.22

application. A brief preliminary screening was designed to test two properties, the long-range (spool-to-spool) and the short-range (tap-to-tap) homogeneity of I_c. Two other key properties were an adequate length and a usable copper-to-superconductor ratio. These four properties were sufficient to pick the conductor to be the SRM.

The homogeneity of I_c for each candidate was determined by measuring two specimens, one 5 and one 50 meters from the end of the shipping spool of each. Five pairs of voltage taps were placed on each specimen. The length of the specimen measured by each pair of voltage taps was about 2 cm and the centers of the adjacent pairs were separated by about 10 cm. The results of this limited preliminary screening pointed out problems with two of the candidates. Although the problem might not be present throughout, both candidates with a possible problem were eliminated.

In no case were the above mentioned problems identifiable with the choice of a given or measured physical parameter or combination of parameters. So the particular parameters for each sample will not be identified. Only the values will be listed here, nonrespectively, for completeness. The wire diameters were: 0.40, 0.51, 0.51, 0.51, and 0.64 mm. The number of superconducting filaments were: 54, 54, 60, 126, and 180. The approximate superconducting filament diameters were: 23, 26, 34, 42, and 50 μm. The copper-to-superconductor ratios were: 1.4, 1.6, 1.8, 1.9, and 2.0 to 1.

The results of the tests on short and long range homogeneity for all five conductors are summarized in Figs. 1 and 2. Figure 1 is a plot of the percentage difference between I_c of the two specimens for each sample at various magnetic fields. Notice that the differences for each sample are about the same at all magnetic fields measured. Sample 4 had a large shift in I_c. Another specimen of this sample, 150 m into the spool, was then measured. The I_c of this specimen had decreased another 1.1% from the value of the 50 m specimen. This may have been due only to an end effect, but this trend did not look good, so sample 4 was eliminated. Figure 2 is a plot of the percentage difference between the measured I_c on the center voltage tap and that of the other four taps for both the 5 and 50 m specimen of each sample. The expected tap profile due to the magnetic field profile has been corrected. Notice that sample 5 seems to have substantially larger variations and, for this reason, was eliminated.

The other two key properties were then used to select the one conductor to be the SRM. One of the remaining candidates was eliminated because the length delivered was considered to be too short (less than 400 m) and an adjacent spool could not be obtained. One of the two candidates left had the lowest copper-to-superconductor ratio (1.4:1). It was eliminated even though the low ratio did not seem to adversely affect the I_c determination, but it could affect the usage of the SRM throughout the many kinds of testing anticipated for an SRM. This left one conductor for further testing.

PROVISIONAL DATA AND STATISTICAL ANALYSIS

Critical current measurements were performed on the chosen SRM. Specimen spools were selected by a systematic sampling procedure to assure that spools were selected along the whole length of wire. No trend or cycle related to distance along the wire was found.

The critical current data were obtained by placing three pairs of voltage taps on each specimen. The length of wire measured by each tap was about 2 cm and the centers of adjacent pairs were separated by about 20 cm. At magnetic fields of 2, 4, 6, and 8 T, two repeat determinations of I_c on each tap were recorded, producing a total of six measurements on each specimen at given (controlled) temperature. Data have been collected from nine spools equally spaced along the length of wire. The physical theory on the shape of the V-I curve and dependence on the weak

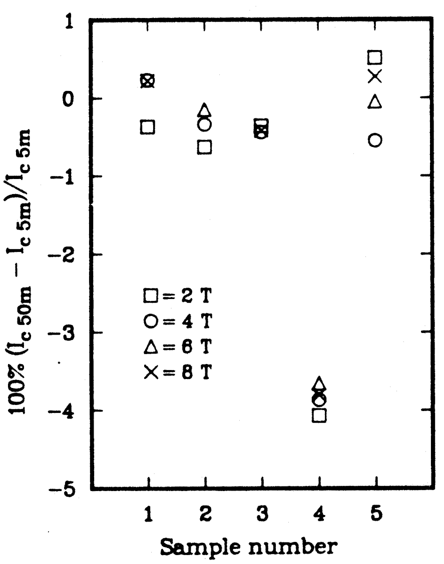

Fig. 1. The percentage change in I_c between the 5 and 50 m specimens of each sample, long range homogeneity.

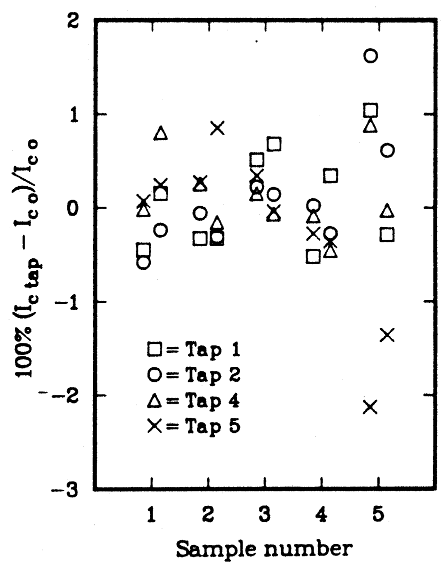

Fig. 2. Short range homogeneity of the 5 and 50 m specimens of each sample (percentage differences relative to center tap).

link suggests that critical current measurements may be nearly summable on a logarithmic scale. Therefore, the data were first transformed to estimate the quantities of interest, and then results on the adjusted scale were converted to the original units of measurement. In the discussion to follow, the estimates of critical current are, in fact, geometric means of the original data because they are derived from simple averages of data on the logarithmic scale.

Critical current measurements at a given field strength can be represented by the statistical model given by,

$$Y_{ijk} = \mu + D_i + T_{ij} + e_{ijk} \qquad \begin{cases} i = 1,\ldots,9; \\ j = 1,\ 2,\ 3; \\ k = 1,\ 2, \end{cases}$$

where Y_{ijk} is the logarithm of the measured critical current for the k-th repeat determination at the j-th tap on the i-th sample spool. Each measurement is thought of as the sum of four components: the mean critical current, μ; a long-range inhomogeneity, D_i, corresponding to distance along the wire; short-range inhomogeneity, T_{ij}, corresponding to the particular tap location on a given spool; and a random measurement error, e_{ijk}. The long- and short-range inhomogeneity terms are considered to be random deviations from the mean critical current for any given spool and tap location. Both the D_i's and T_{ij}'s are assumed to follow Gaussian probability distributions with mean zero and variances σ_D^2 and σ_T^2, respectively. The measurement errors, e_{ijk}, are assumed to have variance σ^2.

An important question for certification is whether all the available spools for this SRM can be treated as identical. The components of variance associated with the long- and short-range material variability of the wire provide a quantitative measure of inhomogeneity in the critical current SRM, so estimates of each type of variation were obtained. The estimated standard deviations, expressed as a percentage error in the critical current at each of the magnetic fields, are given in Table 2. The table illustrates that long-range inhomogeneity is more evident at 2 T and decreases with increasing magnetic field.

Because the critical current measurements revealed substantial long- and short-range variation, the uncertainty statement for the reported critical current at each magnetic field is a statistical tolerance interval. This statistical procedure allows for the observed variation in critical current by estimating limits for the critical current of individual spools, rather than limits on the overall average critical current of all spools.

Table 2. Estimated Long- and Short-Range Inhomogeneity Expressed in Percent Error in I_c at 4.07 K and 0.2 μV/cm.

Magnetic Field	Spool-to-Spool Standard Dev. ($\hat{\sigma}_D$)	Taps within Spool Standard Dev. ($\hat{\sigma}_T$)	Total $(\hat{\sigma}_D^2 + \hat{\sigma}_T^2)^{1/2}$
2	0.41	0.33	0.53
4	0.29	0.26	0.39
6	0.19	0.25	0.31
8	0.18	0.25	0.30

A tolerance interval for the distribution of critical currents for a length of wire of about 2 cm has the form, exp $[\bar{Y} \pm K S_Y]$, where $\bar{Y}$ is the sample mean of all measurements on the logarithmic scale, S_Y is the estimated standard deviation of a single measurement, and K is usually taken from tables such as in Weissberg and Beatty.[7] The value of K = K(N, f, P, γ) depends on:

(1) N: the effective number of observations for $\bar{Y}$;
(2) f: the degrees of freedom associated with S_Y;
(3) P: the proportion of critical current measurements to be covered; and
(4) γ: the probability level associated with the tolerance interval.

The estimated superconducting critical currents for an electric field criterion 0.2 μV/cm for a wire length of 2 cm are given in Table 3. The uncertainty of the reported value, ignoring systematic errors, is the statistical tolerance interval constructed such that it should cover 99% of critical current determinations with probability 0.95. The tolerance limits in Table 3 are expressed in terms of the resultant percentage error at each magnetic field and apply to a single measurement on any given spool for a length of wire of about 2 cm. Uncertainties for wire lengths greater than 2 cm are expected to be less than those in Table 3. The experimental systematic error and the tolerance limit are summed to give the total uncertainty and are all expressed as percent error in I_c.

CONCLUSIONS

A carefully controlled acquisition and analysis system was developed to measure I_c. Two of the five candidate conductors displayed short- or long-range inhomogeneity that made them seem unfit for use as an SRM. The conductor chosen for the SRM had substantial long- and short-range variation in I_c; therefore the uncertainty

Table 3. Provisional Results at 4.07 K and 0.2 μV/cm.

Magnetic Field (T)	Critical Current (A)	Total Uncertainty (%)	Tolerance Limits (%)	Systematic Error (%)
2	301.34	2.40	2.07	0.33
4	193.87	1.86	1.52	0.34
6	130.65	1.53	1.15	0.38
8	75.58	1.68	1.12	0.56

statement was calculated using statistical tolerance limits. The resulting total uncertainty was within the objective of the study.

ACKNOWLEDGMENTS

The help and support of the Superconductors and Magnetic Materials Group, especially F. R. Fickett and J. W. Ekin, are gratefully acknowledged. Suggestions on the content and presentation of this paper from W. S. Liggett, Jr. of the Statistical Engineering Division are appreciated.

REFERENCES

1. J. D. Cali, T. W. Mears, R. E. Michaelis, W. P. Reed, R. W. Seward, C. L. Stanley, H. T. Yolken, and H. H. Ku, "The Role of Standard Reference Materials in Measurement Systems," NBS Monograph 148, National Bureau of Standards, Washington, D.C. (1975) 54 pp.
2. L. F. Goodrich, D. F. Vecchia, E. S. Pittman, A. F. Clark, and J. W. Ekin, to be published.
3. L. F. Goodrich and F. R. Fickett, Critical current measurements: A compendium of experimental results, Cryogenics 22:225-41 (1982).
4. L. F. Goodrich, J. W. Ekin, and F. R. Fickett, Effect of twist pitch on short-sample V-I characteristics of multifilamentary superconductors, Adv. Cryog. Eng. 28:571-80 (1982).
5. J. W. Ekin, Mechanical and properties and strain effects in superconductors, in: "Superconductor Materials Science: Metallurgy, Fabrication, and Applications," S. Foner and B. B. Schwartz, eds. (Plenum Press, New York, 1981) p. 455.
6. J. W. Ekin, private communication.
7. A. Weissberg and G. H. Beatty, Tables of tolerance - Limit factors for normal distributions, Technometrics 2:483-500 (1960).