

Standard reference devices for high temperature superconductor critical current measurements*

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Obtaining repeatable critical current measurements for a high temperature superconductor (HTS) is a challenging task, since HTSs are highly susceptible to degradation due to mechanical stress, moisture, thermal cycling and aging. This paper discusses the development of a high temperature superconducting standard reference device (SRD) to address these measurement concerns and gives preliminary data on its characteristics. An SRD is an HTS specimen that has had its critical current I_c non-destructively evaluated. Because HTSs are sensitive to mechanical alterations, minor changes in sample preparation or mounting procedure could yield large changes in the measured critical current. Preliminary data on SRDs made using Bi-based oxide tapes (2212) with an Ag substrate are presented. Differences between two consecutive measurements of I_c can typically change by 40%; these deviations have been reduced to $\approx 4\%$.

Keywords: high T_c superconductors; critical currents; measuring methods

Accurate measurements of sample voltage, current, temperature and magnetic field do not necessarily imply that the measured I_c will be accurate: the critical current measurement is dominated by concomitant factors such as sample preparation, mounting and measurement methodology. In 1989, an interlaboratory comparison of critical current measurements was conducted¹ in which each participant was given a few specimens from the same batch. Each participating laboratory measured and reported the I_c of the specimens. The reported critical current densities J_c had high variability: J_c values were distributed relatively uniformly from less than 100 to 1000 A cm⁻² for bulk YBCO in liquid nitrogen and zero applied magnetic field. This high variability was probably not primarily due to the specimen inhomogeneity of I_c , since it was determined to be $\approx 10\%$, nor was it primarily due to errors in magnetic field or temperature calibration.

These results do not necessarily indicate that the I_c measurements were inaccurate or incorrect; the specimens may have undergone various amounts of degradation during the measurement, thus changing the I_c of the conductor. The results of this experiment and our own experience measuring high temperature superconductors (HTSs) stressed the need for standardization of critical current measurement methods to reduce variability, the necessity of standard reference devices and a return to the more classical method of performing interlaboratory comparisons in which an individual specimen is meas-

ured by more than one laboratory. Various approaches to conducting interlaboratory comparisons of critical current measurements are discussed in reference 2.

We have dedicated part of our research at NIST to developing a set of high temperature superconductor standard reference devices (HTS-SRDs) to address these measurement concerns. These devices would be used in the same spirit as the superconductor critical current simulator³ and low temperature superconductor (LTS) standard reference materials such as SRM 1457⁴: to determine the integrity of the critical current measurement system and measurement methodology. A typical set of SRDs would include a superconductor simulator, a fully instrumented (current and voltage leads attached) HTS sample and an uninstrumented HTS sample. The I_c of a particular SRD will be well characterized by the control laboratory so that independent laboratories can measure the I_c and compare their measurement with the expected value. This methodology bypasses the problem of I_c inhomogeneity, since each specimen will be well characterized. This comparison will indicate whether there are problems with the measurement. Obtaining an accurate critical current measurement on an SRD is a necessary but not sufficient condition for making good measurements on other samples.

A superconducting SRD not only tests the measurement instrumentation, technique and analysis as the superconductor simulator does; it also tests the effects of sample preparation, magnetic field calibration, contact resistance and other parameters that are unique to superconducting samples. Each SRD in the set has a different level of instrumentation to delineate possible

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sources of I_c measurement variability. Because the SRD will undergo multiple measurements, the device's characteristics must be relatively constant through thermal cycling, shipping and handling.

The motivation for developing the SRD is three-fold:

- 1 to identify the present level of measurement variability and the typical sources of measurement variability;
- 2 to offer insight into reducing the variability; and
- 3 to develop standard measurement methodology for non-destructively determining the critical current of HTS materials so that sample contamination and alteration for subsequent measurements is reduced; the repeatability of critical current measurement for HTSs must be demonstrated.

The experimenter's sample preparation and mounting techniques are studied implicitly during the analysis of the critical current measurement, since they affect the measured I_c and also the appearance of the second derivative (d^2V/dI^2) and n -value curves⁵ of the voltage-current ($V-I$) characteristic. These are viable diagnostic tools for evaluating the merit of a superconductor; however, they are non-trivial measurements: the d^2V/dI^2 measurement is particularly difficult if the sample has a high I_c or high normal state resistance.

Experimental apparatus: pressure contacts and warming chamber

The technologies presented in this section were developed to reduce measurement variability and improve the repeatability of the critical current measurement for the development of SRDs. For single measurements (no repeat determinations), pressure contacts and warming chambers may not be needed.

Pressure contacts

Pressure contacts for the current and voltage taps were used to reduce solder contamination and thus enhance the non-destructive evaluation (NDE). Another advantage of pressure contacts over soldering is that the sample does not experience temperature excursions due to the soldering iron. However, mechanical effects may continue to affect the sample. The contacts were designed to touch the Ag surface of the conductor without excessively damaging the sample. The pressure contacts described here generally have ohmic (linear $V-I$) characteristics. The contact resistance of a typical pressure contact was measured as a function of current. The resistance at 120 A was within 1% of the resistance at 10 A.

The pressure contacts for the current taps were made using a Be-Cu coil spring that applies ≈ 22 N (5 lb) onto the conductor. The voltage taps were also made using Be-Cu leaf springs with a contact force of ≈ 0.8 N (3 oz). Be-Cu springs were chosen because they retain their elasticity at cryogenic temperatures and are non-magnetic. Pure In was soldered to the surface of the contacts, which were subsequently pressed onto the Ag surface. Pure In was used since it is likely to cold-weld to the Ag surface, thus reducing the contact resistance. Although the contact resistance achieved by using pressure contacts (typically $0.2-5 \mu\Omega$ for contacts of $\approx 2 \times 3$ mm) is not as low as for soldering, it does not dissipate signifi-

cant power for currents below 150 A. These powers are manageable assuming the sample is immersed in liquid helium. Solder contacts are required if the sample is cooled in helium gas. The lower contact resistances were achieved by applying an additional force of 53 N (12 lb) to the current contacts at room temperature and then removing this force. The coil spring was relied upon to retain some force through the measurement.

In Figure 1 the sample is orientated so that the Lorentz force is directed into the sample holder, thus decreasing the possibility of sample damage. Nb₃Sn-based tapes were used for the current leads since our measurements are done at 4.0 K and the tape geometry was well suited for pressure contacts.

The coil spring pushes on an insulated yoke that presses on the top of both current leads, thus pressing the In solder onto the Ag surface of the HTS specimen. The In is soldered onto the current contact and then textured by rolling a knurled tool over it, thus creating a number of well defined ridges. Since the local pressure on these ridges is high, cold welding is promoted. Texturing results in a relatively constant solder thickness. Texturing also breaks up any surface oxide and allows for deformation to accommodate any slight misalignment of the pressure fixture. After the In is textured, the excess is trimmed off so that the region of In is only positioned over the specimen and does not touch other parts of the apparatus.

Valved warming chamber

A valved chamber was designed in which to warm the superconductor from cryogenic temperatures to room temperatures to reduce the effects of airborne moisture condensation on the specimen during the warm-up period. Condensation is known to corrupt the critical current measurement.

The valved warming chamber is part of a tubular sample test fixture. The test fixture consists of a long tube with room-temperature connections at one end for current and voltage leads, along with other instrumentation. The opposite end of the fixture has a specimen mounting site. This tube slides through the warming chamber which has an adjustable O-ring seal at the top and a gate valve at the bottom. This allows the sample to be

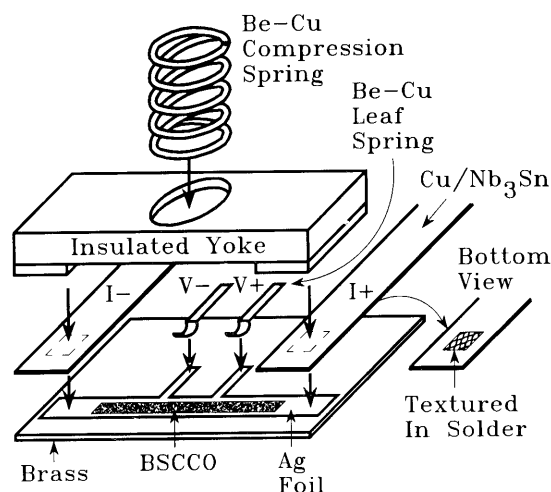


Figure 1 Exploded view of pressure contact design with bottom view of one of the current contacts showing textured In

withdrawn from the liquid helium into the warming chamber which is subsequently sealed off with the gate valve. The chamber is heated until the sample is near room temperature so that the sample is not exposed to air while it is cold. The chamber has a relief valve to control the pressure of the expanding gas. The engagement of the test fixture through the O-ring can be adjusted so that the sample is in the centre of the magnet during testing.

Standard reference device concept and design

One of our SRD design objectives was to develop a non-destructive test methodology that minimally alters the SRD so that the variation in I_c measurement as a function of non-intrinsic conductor parameters such as thermal cycling, sample mounting procedures and cooling rate can be characterized. The critical current of HTSs, unlike their LTS counterparts, is a strong function of these non-intrinsic parameters. These factors will thus be delineated from intrinsic properties. The SRD electrical design target is to have an I_c of 100 A at a temperature of 4 K with zero applied field and a large surface area exposed to liquid helium to facilitate sample cooling. Preliminary results are quite favourable: $\Delta I_c \approx \pm 4\%$ is possible and would not significantly add to the uncertainties achieved in LTSs⁶. In this and subsequent discussions, ΔI_c is defined as the per cent difference between two consecutive measurements.

We have already developed and are testing two SRDs: the first is an uninstrumented device, which would be used by those experimenters who want to test the effects of their current and voltage tap mounting along with their measurement system and measurement technique. The second SRD is a fully instrumented device, meaning that the current and voltage leads are soldered onto the sample. Experimenters would use such a device to test only their measurement system and technique, but not their mounting procedures.

The designs proposed here are not final; they represent preliminary ideas on developing an SRD. Other designs such as fully encapsulated modules are also possibilities for reference devices. These devices would be constructed so that the superconductor is fully encapsulated in an Ag sheath. This sample would have an advantage over the SRD proposed here since the sample would be less susceptible to aging and moisture degradation. However, the fully encapsulated device would not test these effects, which may be present in superconductor measurement experiments. A wide range of SRD designs may be necessary to fully characterize the spectrum of measurement conditions.

Uninstrumented standard reference device

Experimenters would measure the characteristics of the uninstrumented SRD after they apply their current and voltage leads, and compare their results to the expected value. This requires that a control laboratory perform NDE of the I_c of each specimen before the independent laboratory. If there is a significant difference between these values, it could indicate that the sample's characteristics were altered during the mounting of the current and voltage taps. Figure 2 shows the uninstrumented SRD

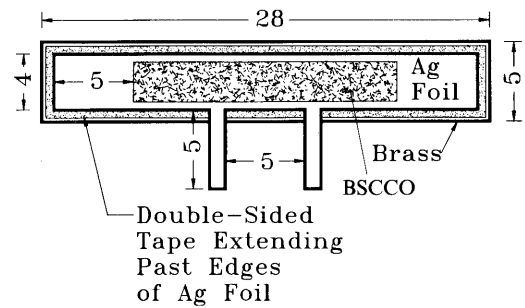


Figure 2 Uninstrumented SRD, featuring well defined Ag voltage tap locations (all dimensions are in mm)

and features well defined Ag voltage tap locations (fins). The NDE specimen is mounted to a brass plate ($0.05 \times 0.5 \times 2.8$ cm) and is electrically insulated from the plate. Evidence that the crucial step of NDE did not alter the I_c of the conductor is presented later in this paper.

Fully instrumented standard reference device

The fully instrumented SRD has current and voltage taps already soldered to the sample. Figure 3 shows a prototypical design for the fully instrumented SRD and contains a brass plate (0.05×1.7 cm \times 2.8 cm) on which the Ag foil is mounted. The specimen is electrically insulated from the plate. A non-conducting plate such as fibreglass-epoxy composite could also be used. Many features shown in Figure 3 will be carried on to subsequent fully instrumented SRD designs.

The fully instrumented SRD has brass tubes mounted on the base plate to serve as holders for the current and voltage taps. These tubes provide strain relief for the current and voltage leads and allow for longer current leads to improve thermal isolation of the specimen from the soldering iron. This design uses round insulated Cu/Nb-Ti wires for current leads to reduce power dissipation at 4 K up to fields of 6 to 8 T and to retain the flexibility

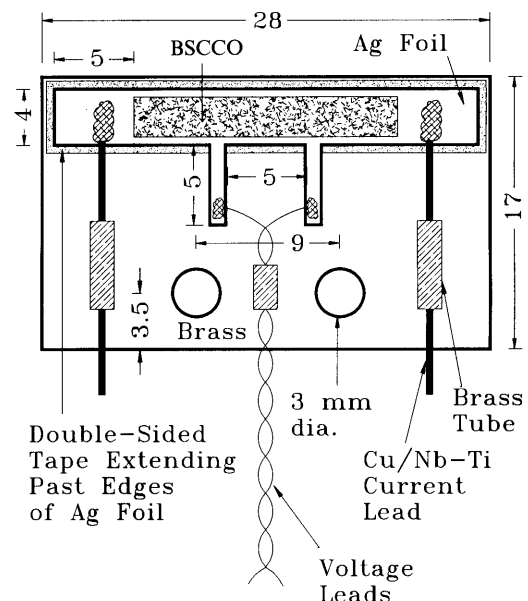


Figure 3 Fully instrumented SRD, featuring brass tubes mounted on brass backing for strain relief for current and voltage leads (all dimensions are in mm)

of the leads necessary to reach the current bus bar on a typical apparatus.

Preliminary results on standard reference devices

The preliminary results indicate that it is possible to achieve high precision and repeatability in the measurement of I_c on HTS-SRDs. These devices are being characterized by their measured change in I_c after thermal cycling and aging, as well as their performance as a function of cooling rate, soldering and desoldering. Two types of superconducting specimens with different geometries were used to test the reference device design concept. For brevity, only three of the many experiments used to characterize the design concept are discussed here. All critical current measurements were made on a computer driven data acquisition system⁷.

The first specimen tested was a thick film Bi-based oxide superconductor (Bi-Sr-Ca-Cu-O: 2212, BSCCO: 2212 for short) with an Ag substrate (25 μm thick) on the top and bottom of the tape (edges of BSCCO were exposed). The Ag and BSCCO were not patterned in the same manner as shown on *Figures 2* and *3*; rather, the Ag and BSCCO edges were flush with the overall specimen dimensions. The specimen dimensions were 84 μm \times 8 mm \times 30 mm. This is a more typical specimen geometry for long length production. This specimen was mounted using epoxy to a fibreglass-epoxy composite board of dimensions 0.05 \times 1.7 \times 3.0 cm. The cross-sectional area of the superconductor was \approx 34 μm \times 8 mm (0.27 mm²). J_c was \approx 350 A mm⁻² at 0 T and 4 K with an electric field criterion E_c of 1 $\mu\text{V cm}^{-1}$. Other samples on one Ag substrate and a thinner layer of superconductor had a J_c of \approx 2400 A mm⁻² at 0 T and 4 K with an E_c of 1 $\mu\text{V cm}^{-1}$. However, the I_c of 245 A was too high for the d^2V/dI^2 analysis. This specimen was a prototypical uninstrumented SRD conductor, meaning that it was not preinstrumented. The I_c of the specimen was measured in the following sequence of experiments (A, B, C) at E_c values of 1 $\mu\text{V cm}^{-1}$ and 10 $\mu\text{V cm}^{-1}$ with an applied magnetic field from 0 to 12 T:

- A Pressure contacts were applied to the specimen for current and voltage taps. The positions of these contacts were noted so that subsequent measurements could be made with the voltage taps in the same position. The pressure contacts should be mounted in nearly the same position from run to run in order to ensure that the same portion of the conductor is being measured; inadvertent misplacement of the voltage taps could yield different critical currents. This section of the experiment simulates the control laboratory performing the first measurement on the conductor to obtain the I_c .
- B The specimen was warmed in the valved warming chamber to room temperature. The pressure contacts were dismantled and remounted within 0.5 mm of the original mounting site. Again, I_c was measured at the same criteria as above. Run B tests whether or not the measurement performed in A is non-destructive. The change in I_c obtained from a repeat run with a remount is used to infer how much the critical current may have changed before it was measured in run A.

- C In the final run, the current and voltage taps were soldered to the specimen using In-Sn eutectic solder. This solder was chosen because it has a relatively low melting point (118°C) and would thus reduce the temperature extremes experienced by the specimen⁸. In this section of the experiment, the I_c measurement of the independent laboratory is simulated.

Because of the inherent instabilities of HTSs, we expected the overall ΔI_c from run A to run C to be significantly more than the observed value of 3.9%. Based on these results, it is now possible to estimate the accuracy of our I_c measurements at $\pm 5\%$ with a precision of $\pm 1\%$. This low variation indicates that the effect of pressure contact remounting compounded with thermal cycling and soldering has a small effect on the specimen's electrical characteristics. The results of this experiment show that a future interlaboratory comparison of I_c measurements of HTSs is feasible and that the effects of application of pressure contacts, thermal cycling and thermal excursions due to soldering can be reduced to manageable levels. *Figure 4* shows the critical currents for runs A, B and C at 4 K in applied magnetic fields of 0 and 4 T. At a magnetic field of 0 T, the overall reduction in I_c was $\approx 3.9\%$, while at 4 T, the reduction was of the order of 1.2%. Data at other magnetic fields (0.1, 0.2, 0.5, 1, 2, 8 and 12 T) showed similar trends. The largest value of ΔI_c was observed at the lower magnetic fields. However, measurements at zero field were indicative of changes at high fields.

The second experiment was designed to test the SRDs and the measurement methodology as a function of thermal cycling and cooling rate. The sample geometry is shown in *Figure 2*. The conductor used was a Bi-based oxide (2212) superconductor specimen with an Ag substrate mounted on and insulated from a brass backing.

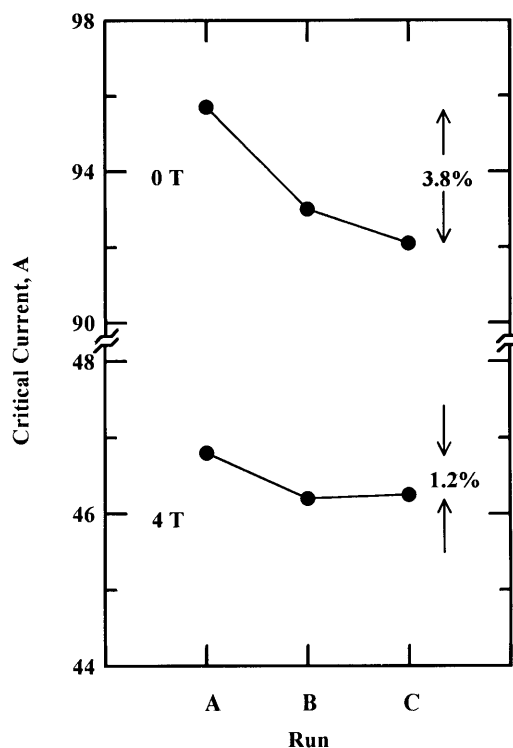


Figure 4 Critical currents for runs A, B and C at 4 K with applied magnetic fields of 0 and 4 T

The cross-sectional area of the superconductor was $\approx 8 \mu\text{m} \times 2 \text{mm}$ (0.016mm^2) with a J_c of $\approx 4500 \text{A mm}^{-2}$ at an E_c of $1 \mu\text{V cm}^{-1}$, a magnetic field of 0 T and a temperature of 4 K. The specimen configuration included a pair of voltage tap 'fins' cut from the Ag substrate to localize the position of the voltage taps. The strip of oxide superconductor does not cover the total area of the Ag so that current and voltage contacts can be made to the top of the Ag and the Ag can be mounted to the backing. Pressure contacts were used for the voltage and current taps.

The specimen was thermally cycled nine times; two of these runs included remounts of the pressure contacts. The specimen was exposed to the laboratory atmosphere during the remounts. In the other seven runs, the sample was thermally cycled from 4 to $\approx 250 \text{K}$ and remained in the helium gas environment of the Dewar. The last run had an extremely high cooling rate: more than 33K s^{-1} . For the high cooling rate run, the specimen was rapidly lowered from room temperature into liquid helium. The overall change in I_c was less than 4% from the first run to the ninth run. Figure 5 is a plot of I_c as a function of the run number for zero applied magnetic field. The monotonic reduction of critical currents shown on Figure 5 indicates that the specimen did not experience significant damage during the entire experiment. The remounts occurred between runs 3 and 4, and runs 4 and 5. The plot indicates systematic sample degradation over the nine runs. The larger changes in critical current occurred after remounts or exposure to a high cooling rate.

Second derivative characteristic and n -value as figures of merit

Figure 6 is a plot of two second derivative curves of the voltage-current characteristics for a Bi-based (2212) oxide specimen with an applied magnetic field of 1 T. The curves correspond to two runs on the same specimen, with \square corresponding to run B, and \circ corresponding to run C. The curves are normalized with respect to the maximum value of the second derivative on run B; the critical currents at 1 and $10 \mu\text{V cm}^{-1}$ are shown for each

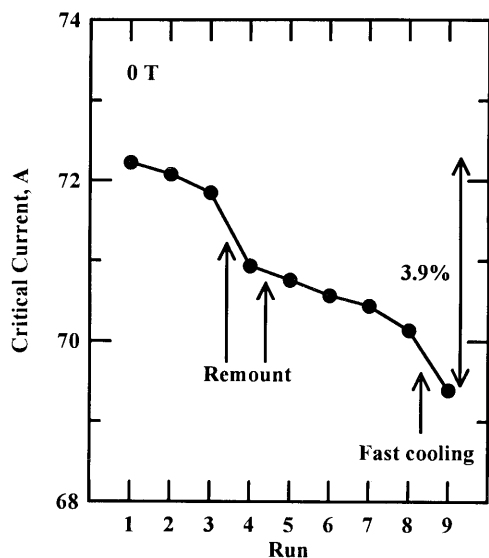


Figure 5 I_c at 4 K as a function of run number for BSSCO (2212) sample with zero applied magnetic field

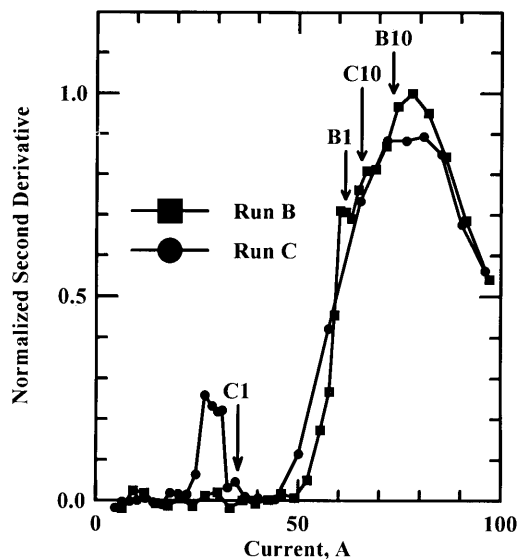


Figure 6 d^2V/dI^2 versus current for BSSCO (2212) sample at 4 K with applied magnetic field of 1 T. The curves correspond to two runs with the same sample: \square , run B; \circ , run C. The critical currents at 1 and $10 \mu\text{V cm}^{-1}$ are shown for each curve, and are coded with the appropriate letter and E_c .

curve. The n -value at low voltage for run B was ≈ 22 . This series of runs used pressure contacts and the sample was remounted between each run. The difference between runs A and B for this sample was relatively small, so it was omitted for clarity.

The second derivative of the voltage-current characteristic could be considered as a distribution of critical current densities⁵. The curve for run B has typical characteristics of a conductor in 'good' condition: a relatively smooth region preceding a well defined peak. The d^2V/dI^2 characteristic for run C shows an anomaly: the curve shifts to lower currents with a sub-peak near 30 A. A transition of this type indicates that the specimen was damaged between runs B and C, or during run C. This damage is also reflected in the 40% reduction in I_c at $1 \mu\text{V cm}^{-1}$. The I_c only fell by 10% at an E_c of $10 \mu\text{V cm}^{-1}$. This figure shows how the second derivative plot can be used as an aid in determining the state of the conductor.

There was evidence that a small crease had formed in the region between the voltage taps on this sample. It is speculated that this specimen damage led to the formation of the peak near 30 A. The n -value calculated using the derivatives of the curve was ≈ 1 between the two peaks in the second derivative characteristic. This indicates that the small part of the specimen contributing to the distribution of low J_c values was ohmic at these currents. The excess current is shunted around this part through the Ag substrate. This sample was not mounted to a backing. It was most likely damaged during the remounting. A backing of fibreglass-epoxy composite or brass with a thickness of merely 0.05 cm makes the samples much easier to handle.

As the conductor changes due to aging, mechanical damage and thermal cycling, the critical current reduces and the second derivative characteristic exhibits a higher population of critical current densities at lower currents. The shape of the V - I curve is a good indicator of the state of the conductor. A sharp, well defined peak in the

d^2V/dI^2 characteristic usually indicates that the sample is in good condition. Such a peak has not yet been observed on a damaged sample. Thus, the second derivative of the voltage-current characteristic can be used as a tool to identify whether or not a sample has undergone damage during an interlaboratory comparison of critical currents. The n -value characteristic could also be used as such an aid⁵; a high n -value indicates a well defined transition. The n -value can be computed from the data taken during the critical current measurement. Thus, it is a convenient figure of merit.

Discussion

The design of the superconductor SRD is relatively independent of the type of sample being used. Thus, an HTS such as a Bi-based oxide conductor could be used, or an LTS such as Cu/Nb-Ti could also be used for an LTS-SRD. In a typical interlaboratory comparison of critical currents, three reference devices could be used: the simulator, an LTS-SRD and an HTS-SRD. The type of LTS and HTS reference device (uninstrumented SRD, fully instrumented SRD) could be chosen by the control laboratory depending upon which experimental parameters need to be tested. The measurement process must reflect the future application of the superconductor.

Some details of the sample fabrication process may affect the suitability of samples as SRDs. Extreme inhomogeneity of the I_c within a specimen would increase the sensitivity to voltage tap placement. The phase purity of the oxide superconductor may increase its sensitivity to aging and moisture exposure. The amount of Ag that migrates into the superconductor during sample fabrication may affect the sensitivity to mechanical failures of the sample, such as cracking due to handling or differential thermal contraction. Thus, there is a need to verify that a particular sample is suitable for use as an SRD.

The SRDs shown in this paper were constructed with many design considerations to improve the repeatability of the critical current measurement. The SRD would typically be measured twice or at most three times: the central laboratory would measure the critical current and send it to a participating laboratory which would again measure it. The participating laboratory could request that the central laboratory measure the I_c again, thus leading to a possibility of three measurements. Preliminary data suggest that if three measurements are planned, the participating laboratory should have a valved warming chamber; otherwise they do not need one. Further investigations may reveal limitations on the number of thermal cycles, the cooling rate and exposure of the cold sample to the atmosphere.

The technologies employed in the design and construction of the SRDs were created to aid in obtaining low measurement variability. Samples bonded to a fibreglass-epoxy plate with epoxy and samples bonded to a brass plate with double-sided adhesive tape both showed satisfactory critical current measurement results. It may be possible to obtain repeatable measurements on a sample without a backing. However, there would be much more uncertainty in the measurement. It was found that mounting the voltage taps to voltage tap fins yielded satisfactory results, as did mounting the voltage taps directly to the sample. The voltage tap fins were antici-

pated to yield more repeatable results since they offer well defined voltage tap positions, and also isolate the sample from the voltage tap wire connections somewhat. This isolation may be important when solder is used to affix the voltage taps to the specimen.

User-specified standard reference device

The user-specified SRD could be constructed according to a specific set of design parameters as given by a user. Users would determine which experimental parameters need to be tested in the context of their experiment and would submit a proposal to the control laboratory. The control laboratory could construct and measure a custom device and return it to the user. The user would measure the I_c of the device and compare it to the expected value.

The design parameters given by the user could vary from dimensional changes in the conductor to fit a particular test fixture, to changes in the type of superconductor used in the SRD. It may not be possible to construct a viable SRD from any superconductor sample.

Conclusions

An SRD is an HTS specimen that has a well characterized critical current. Two SRD configurations are employed to help identify uninstrumented sources of measurement variability: an uninstrumented configuration allows the user to instrument the specimen in any manner, whereas the fully instrumented configuration has current and voltage leads attached to the specimen to remove a number of variables from the comparison. The applications of the SRDs are broad: the superconductor industry as well as research institutes could use them to assess measurement accuracy, determine the sources of measurement variability and reduce the overall variability of the critical current measurement.

The prototype designs and measurement techniques discussed here for the HTS-SRDs have yielded changes in critical current $< \pm 4\%$. This indicates that an interlaboratory comparison of critical current measurements is feasible in the near future. Various measurement techniques that reduce the variability of the critical current measurement have also been explored. Pressure voltage and current contacts and a sealed warming chamber are useful technologies for NDE of the critical current of the conductor, which is a vital step in this characterization.

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