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INTERNATIONAL STANDARD

NORME INTERNATIONALE

colour inside

Superconductivity –
Part 23: Residual resistance ratio measurement – Residual resistance ratio of Nb superconductors

Supraconductivité –
Partie 23: Mesurage du rapport de résistance résiduelle – Rapport de résistance résiduelle des supraconducteurs de Nb

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IEC Central Office
3, rue de Varembé
CH-1211 Geneva 20
Switzerland

Tel.: +41 22 919 02 11
info@iec.ch
www.iec.ch

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SUPERCONDUCTIVITY –

Part 23: Residual resistance ratio measurement – Residual resistance ratio of Nb superconductors

FOREWORD

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The text of this International Standard is based on the following documents:

FDIS	Report on voting
90/400/FDIS	90/403/RVD

Full information on the voting for the approval of this International Standard can be found in the report on voting indicated in the above table.

This document has been drafted in accordance with the ISO/IEC Directives, Part 2.

A list of all parts in the IEC 61788 series, published under the general title *Superconductivity,* can be found on the IEC website.

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INTRODUCTION

High-purity niobium is the chief material used to make superconducting radio-frequency cavities. Similar grades of niobium may be used in the manufacture of superconducting wire. Procurement of raw materials and quality assurance of delivered products often use the residual resistance ratio (RRR) to specify or assess the purity of a metal. RRR is defined for non-superconducting metals as the ratio of electrical resistance measured at room temperature (293 K) to the resistance measured for the same specimen at low temperature (~4,2 K). The low-temperature value is often called the residual resistance. Higher purity is associated with higher values of RRR.

Niobium presents special problems due to its transformation to a superconducting state at ~9 K, so DC electrical resistance is effectively zero below this temperature. The definition above would then yield an infinite value for RRR. This document describes a test method to determine the residual resistance value by using a plot of the resistance to temperature as the test specimen is gradually warmed through the superconducting transition in the absence of an applied magnetic field. This results in a determination of the residual resistance at just above superconducting transition, ~10 K, from which RRR is subsequently determined.

International standards also exist to determine the residual resistance ratio of superconducting wires. In contrast to superconducting wires, which are usually a composite of a superconducting material and a non-superconducting material and the RRR value is representative of only the non-superconducting component, here the entire specimen is composed of superconducting niobium. Frequently, niobium is procured as a sheet, bar, tube, or rod, and not as a wire. For such forms, test specimens will likely be a few millimeters in the dimensions transverse to electric current flow. This difference is significant when making electrical resistance measurements, since niobium samples will likely be much longer than that for the same length-to-diameter ratio as a wire, and higher electrical current may be required to produce sufficient voltage signals. Guidance for sample dimensions and electrical connections is provided in Annex A. Test apparatus should also take into consideration aspects such as the orientation of a test specimen relative to the liquid helium surface, accessibility through ports on common liquid helium dewars, design of current contacts, and minimization of thermal gradients over long specimen lengths. These aspects distinguish the present document from similar wire standards.

Other test methods have been used to determine RRR. Some methods use a measurement at a temperature other than 293 K for the high resistance value. Some methods use extrapolations at 4,2 K in the absence of an applied magnetic field for the low resistance value. Other methods use an applied magnetic field to suppress superconductivity at 4,2 K. A comparison between this document and some other test methods is presented in Annex A. It should be noted that systematic differences of up to 10 % are produced by these other methods, which is larger than the target uncertainty of this document. Care should therefore be taken to apply this document or the appropriate corrections listed in Annex A according to the test method used.

Whenever possible, this test method should be transferred to vendors and collaborators who also perform RRR measurements. To promote consistency, the results of inter-laboratory comparisons are described in Annex C.

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SUPERCONDUCTIVITY –

Part 23: Residual resistance ratio measurement – Residual resistance ratio of Nb superconductors

1 Scope

This part of IEC 61788 addresses a test method for the determination of the residual resistance ratio (RRR), r_{RRR}, of cavity-grade niobium. This method is intended for high-purity niobium grades with $15 < r_{RRR} < 600$. The test method should be valid for specimens with rectangular or round cross-section, cross-sectional area greater than 1 mm^2 but less than 20 mm^2, and a length not less than 10 nor more than 25 times the width or diameter.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60050-815, *International Electrotechnical Vocabulary – Part 815: Superconductivity* (available at: www.electropedia.org)

3 Terms and definitions

For the purposes of this document, the terms and definitions given in IEC 60050-815 and the following apply.

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3.1
residual resistance ratio
RRR
ratio of resistance at room temperature to the resistance just above the superconducting transition

$$r_{RRR} = R_1 / R_2 \quad (1)$$

where R_1 is the resistance at 293 K and R_2 is the resistance just above the superconducting transition, at ~10 K.

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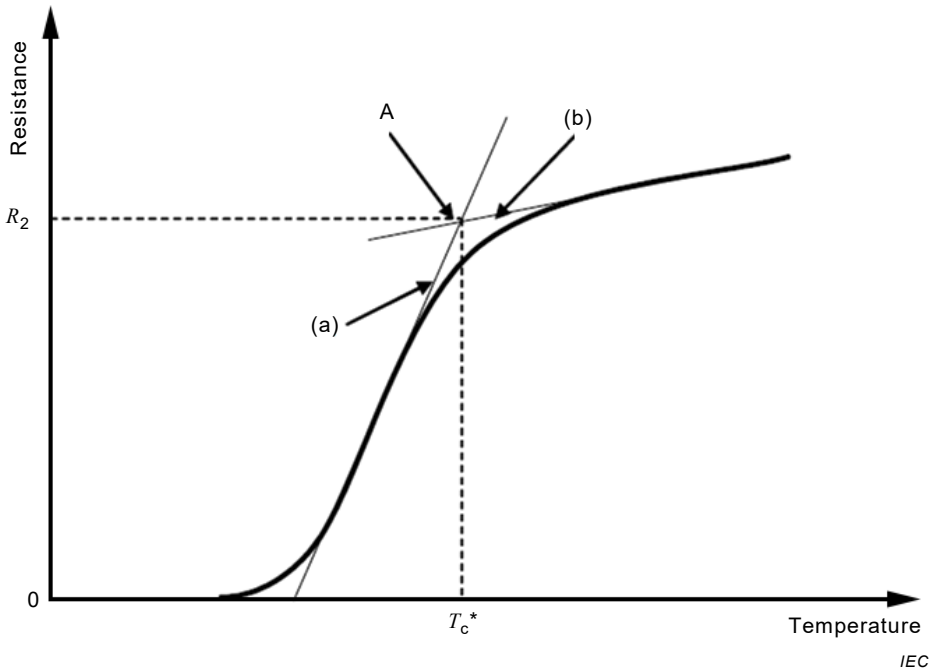

Figure 1 – Relationship between temperature and resistance near the superconducting transition

Note 1 to entry: In this document, the room temperature is defined as 20 °C = 293 K, and r_{RRR} is obtained as follows: Figure 1 shows schematically resistance versus temperature data and the graphical procedure used to determine the value of R_2. In this figure, the region of maximum slope is extrapolated upward in resistance, as shown by line (a), and the region of minimum slope at temperatures above the transition temperature is extrapolated downward in temperature, as shown by line (b). The intersection of these extrapolations at point A determines the value of R_2 as well as a temperature value T_c^*.

Note 2 to entry: The value T_c^* is similar to the transition value defined in [1] [1], and should not be confused with the value defined at the midpoint of the transition, called T_c^* in [2].

Note 3 to entry: Some standards or documented techniques, e.g. [3][4][5], define r_{RRR} with the value of R_1 determined at a temperature other than 293 K, or the value of R_2 determined at a temperature below the superconducting transition. The user of this document should be alert for such differences in definition.

Note 4 to entry: This note applies to the French language only.

4 Principle

The 4-point DC electrical resistance technique shall be performed both at room temperature and at cryogenic temperature. The test may be done either as a function of temperature or as a function of time with increasing temperature.

The relative combined standard uncertainty of this method is 3 % with coverage factor 2.

Measurements shall have the following attributes:

a) Measuring current is sufficiently high to provide voltage signals of the order of 1 µV. For electrical safety, maximum current density should never exceed 1 A mm^{-2}.

[1] Numbers in square brackets refer to the Bibliography.

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b) Contact resistance for current leads is sufficiently low to avoid excessive heating of the sample. Typical cryogenic measurement conditions require power dissipation at contacts to be less than 1 mW.

c) Sample sizes shall be sufficiently large to minimize effects from cutting and handling damage. Typical samples are 1 mm to 3 mm in cross-section dimension and > 5 mm^2 in cross-sectional area.

d) Sample length shall be at least 10 times and not more than 25 times the width or diameter.

Annex A discusses considerations for sample dimensions and measuring current.

5 Measurement apparatus

5.1 Mandrel or base plate

A straight mandrel or base plate shall be used to support the specimen. Possible materials of construction include pure copper, pure aluminum, pure silver, electrical grades of Cu-Zr, Cu-Cr-Zr, Cu-Be, and other copper alloys, electrical grades of Al-Mg, Al-Ag, and other aluminum alloys, and electrical grades of silver alloys. These provide high thermal conductivity and serve to remove thermal gradients during measurement. Care should be taken to insulate the specimen from the mandrel. Possible insulating materials include polyethylene terephthalate, polyester, and polytetrafluoroethylene, which may be applied as foils, tapes, or coatings. Glass-fiber reinforced epoxy or other composite materials with good thermal conductivity at cryogenic temperature may also be used.

The base plate should have a clean and smooth surface finish. There should be no burrs, ridges, seams, or other asperities that may affect the specimen. High purity niobium specimens are soft and are susceptible to indentation by surface flaws, and such indentations may alter the sample and invalidate the resistance measurement.

The mandrel or base plate shall support the entire length and width of the specimen. Mandrel or base plate geometry should not impose a bending strain of more than 0,2 % on the sample.

A thermometer accurate to 0,1 K is helpful but not required. The mandrel or base plate may incorporate a mounting for a cryogenic thermometer directly against the body of the mandrel or base plate and near the center of the test specimen.

Practical base plates are at least 30 mm in length to accommodate assembly of pieces and handling of samples by human hands. Multiple samples may be mounted against a single base plate.

5.2 Cryostat and support of mandrel or base plate

The apparatus shall make provisions for mechanical support of the mandrel or base plate. In addition, such support shall provide electrical leads to carry currents for samples and thermometers, and measure their voltages. For R_1 and R_2 measurements, the support shall permit current to flow through only the sample, so that the entire resulting voltage measured is only that generated by the sample.

The support structure shall permit measurement of both R_1 and R_2 without dismounting or remounting the test specimen. Measurement of R_2 shall require the use of a cryostat, which shall, moreover, integrate with the support.

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The cryostat shall include a liquid helium reservoir at the bottom of a substantial vertical column. A support structure shall accommodate the raising and lowering of the sample into or out of the helium bath. In addition, anchoring of the sample position, either while immersed in liquid helium or suspended above the surface of the liquid at an arbitrary height, shall be provided. Such suspension permits the equilibration of temperature during measurement and slow increase of temperature with height above the helium bath. Alternately, immersion of the sample into the bath followed by reduction of the bath level via boil-off or pressurized transfer can also be used to vary temperature.

A heater may be employed to warm the mandrel or base plate. Care should be taken to distribute the heater along the mandrel and avoid excessive power settings. For instance, a point source of 1 W heat input operating at the center of a 1 cm^2 mandrel upon which a 5 cm sample is mounted could produce thermal gradients of 2,5 K along the sample if the thermal conductivity is 100 W m^{-1} K^{-1}.

Proper cryogenic techniques shall be followed for the construction of the cryostat and apparatus. This includes the use of low thermal conductivity materials such as thin-walled stainless steel tubes, composite materials, ceramics, and insulation, to prevent excessive boil-off due to heat conduction from the surroundings. A can or shield may surround the base plate or mandrel with mounted sample to improve thermal stability. Provisions for pressure relief and vacuum isolation of the liquid helium should be incorporated with the apparatus.

6 Specimen preparation

High-purity niobium is quite malleable, and even the slightest force can produce deformation of the material. Since dislocations are one source of electron scattering, specimen deformation may inadvertently contribute to the residual resistivity and affect the test result. Therefore, special protocols shall be observed when preparing the specimen. Cutting techniques shall avoid heat and strain to the extent possible. Discharge machining, fluid-jet cutting, or low-speed conventional machining are acceptable and widely-used techniques for applications using high-purity niobium. Specimens cut from larger pieces should be protected and immobilized against a support piece during transport. Operations to de-burr samples should be done with great care to not bend, excessively heat or otherwise damage the sample. Light sanding with fine paper is one acceptable approach.

Specimens should be rectangular or circular bars with uniform cross-section. Long sides of the specimen shall be parallel. Any twisting or curvature shall be avoided to ensure that bending or torsion is not applied to the test specimen during mounting to the mandrel or base plate. Specimens that form an arc or a “U” shape are acceptable provided that the entire curvature can be supported on a plane, without applying torsion to the bent specimen.

The specimen shall be clean and have no trace of residues from cutting fluids or any other surface contaminants. Degreasing with solvents, followed by ultrasonic cleaning using a mild water-based detergent, followed by rinsing with distilled or ultra-pure water, then drying in air, is preferred for cleaning residues. Chemical etching to clean the surface poses a risk of introducing contaminants, especially hydrogen and oxygen, and should be avoided. Gentle mechanical polishing of the regions where voltage taps and current leads attach is usually sufficient to remove surface oxides. Coating these regions with indium foil or another metal, for example by evaporation or sputtering, is an acceptable method to protect polished contacts provided care is taken to avoid coating the entire specimen.

The test specimen shall be a single piece and shall not include any joints or splices.

A mechanical method shall be used to affix the test specimen to the mandrel or base plate. Special care shall be taken during the installation and instrumentation of the specimen to ensure that there is no excessive force, bending strain, tensile strain, or torsion applied to the specimen.

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The test specimen shall be instrumented with current contacts near each end of the specimen and a pair of voltage contacts over the central portion between the current contacts (i.e. a 4-point measurement technique). The voltage contacts shall be separated from the current contacts by a distance no smaller than the largest dimension (width, thickness, or diameter) perpendicular to the specimen length.

7 Data acquisition and analysis

7.1 Data acquisition hardware

Modern power supplies can be computer controlled and come with a variety of features that permit remote control of the current output. Use of such power supplies is not required but could greatly enable automation of the data acquisition. Pulsed modes permit application of current only when voltage signals are being acquired, thereby removing heat generated in the sample during the off cycle. If pulsed current application is used, the pulse duration shall include ample periods for voltage signals to settle and be filtered.

Some power supplies incorporate an internal shunt to regulate the output current. If such a power supply is used, the internal shunt shall be calibrated periodically with an external shunt and voltage measurement.

The test set-up may establish an arbitrary baseline voltage U_0, which might be detectable when the sample is in the superconducting state and the power supply is off. The value U_0 can drift over time due to changes in the thermal environment and other effects. More complex hardware includes compensation for drift and automatic nulling such that the time average of U_0 is 0. Digital voltage meters are not required but greatly enhance the data acquisition. Besides compensation for drift and voltage nulling, filtering and internal compensation for thermally-induced voltages can improve the accuracy of the voltage measurement. Filtering should average voltage signals for a time at least as long as the thermal time constant of the apparatus at low temperature, typically of the order of 0,1 s to 10 s. Care should be taken to understand how voltages are corrected for drift and thermal effects. Sensitive voltage meters, especially nanovolt meters, require a pre-amplifier that needs to be at thermal equilibrium, which may require several hours of operation in advance of the measurement.

Data acquisition via computer greatly facilitates the recording and reporting of data.

7.2 Resistance (R_1) at room temperature

The ambient temperature T_1 of the measurement laboratory shall be measured. A specimen current I_1 shall be applied per the requirements in Clause 4. The resulting voltage U_1 shall be recorded together with I_1 and T_1. The resistance shall be determined by

$$R_1 = \frac{U_1}{I_1}\left[1 - 0{,}0037\ \left(T_1 - 293\right)\right] \tag{2}$$

with T_1 in units of kelvin. The coefficient 0,003 7 reflects the experimentally observed rate of change of resistance with temperature given in [5].

7.3 Residual resistance (R_2) just above the superconducting transition

The measurement of R_2 shall be made with the sample still mounted on the mandrel or base plate for the measurement of R_1.

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The specimen shall be placed in a cryostat as specified in 5.2. The specimen shall be slowly lowered into a liquid helium bath and cooled to liquid helium temperature. While a vigorous boil-off of liquid helium will accompany the initial cool down, removal of heat from the mandrel, especially if it is shielded, can require a time period of more than 5 min. Current may be applied, and voltage may be monitored during this period, but no measurement shall be made until the vigorous boil-off of liquid helium has subsided.

After the boil-off rate is suitable for measurements, a voltage measurement U_0 shall be recorded while the sample is immersed in liquid helium. The sample should be in the superconducting state under these conditions. Current I_2 shall then be applied per requirements of Clause 4 and with considerations of Clause 5. Voltage readings shall be acquired for forward and reverse current polarity, U_0^+ and U_0^- respectively. Any differences between U_0, U_0^+, and U_0^- shall be recorded.

The specimen shall then be gradually warmed so that a transition from the superconducting state into the normal state occurs gradually. An apparatus that conforms to Clause 6 will permit gradual warming of the specimen by raising the level of the mandrel above the level of the liquid helium bath, for example. Two voltages U_2^+ and U_2^- shall be measured almost simultaneously with the application of the same measuring current I_2 with forward and reverse polarity, respectively. The current shall not be applied when measurements are not being recorded. The voltage U_2 shall be determined by

$$U_2 = \frac{\left|U_2^+ - U_2^-\right|}{2} \tag{3}$$

where it should be noted that the sign of U_2^- is opposite that of U_2^+; i.e. Formula (3) indicates an average of the two numbers approximately equal in magnitude. A resistance R shall be determined from the voltage by

$$R = \frac{U_2}{I_2} \tag{4}$$

As the sample is warmed, values R shall be recorded as a function of either the temperature T determined by the thermometer attached to the mandrel or base plate, or the time t. Graphical aids and data analysis software are acceptable tools for plotting the resistance versus temperature curve or resistance versus time curve and performing extrapolations.

A resistance versus temperature curve shall thus be obtained as in Figure 1. The resistance versus temperature curve shall be continuously recorded until a temperature of at least 15 K is reached. The resistance versus temperature curve shall be analyzed by drawing a line through the region of steepest slope near the midpoint of the resistance rise, line (a) on Figure 1, and extrapolating this line sufficiently above the value of R recorded at 15 K. A second line shall be drawn through the region of the resistance versus temperature curve above the transition, line (b) in Figure 1, and this line shall be extrapolated to sufficiently lower temperature such that it intersects with line (a). The intersection is labelled as point A in Figure 1. The value of resistance R_2 corresponding to intersection point A shall be recorded, along with the value of temperature T_c^* corresponding to intersection point A.

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7.4 Validation of the residual resistance measurement

The determination of R_2 shall be valid if all the following criteria are met: Interfering voltages shall be such that

$$\frac{\left|U_0^+ - U_0^-\right|}{I_2 R_2} < 3\ \% \tag{5}$$

Thermal drift or scatter shall be such that, for consecutive values U_2^+ and U_2^- recorded with temperature near T_c^*,

$$\frac{\left|U_2^+ + U_2^-\right|}{I_2 R_2} < 3\% \tag{6}$$

The ambient temperature shall be such that

$$283\ \text{K} < T_1 < 303\ \text{K} \tag{7}$$

7.5 Residual resistance ratio

The RRR shall be calculated using Formula (1) and recorded.

8 Uncertainty of the test method

Based on the outcome of inter-laboratory comparison, discussed fully in Clause C.2, a typical uncertainty across laboratories of 0,3 % to 1,3 % has been obtained.

9 Test report

9.1 General

A test report shall be provided to summarize the findings of the RRR test procedure.

9.2 Test information

The following shall be included to record the test information:

a) date and time of the measurement;
b) operator name;
c) version of the procedure followed.

9.3 Specimen information

The following information pertinent to the specimen shall be included in the test report:

a) vendor's heat treatment, fabrication, or other tracking information such as a purchase order number;
b) sheet or piece identification number, if any;
c) specimen shape and orientation relative to the helium bath.

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9.4 Test conditions

The following test conditions shall be included in the test report:

a) room temperature T_1;

b) transport currents I_1 and I_2;

c) voltages U_1 and U_2, noting that U_2 varies with temperature and therefore requires reporting as a table or graph;

d) resistances R_1 and R_2

e) voltages U_0, U_0^+, U_0^- or validation condition (5);

The following additional information may be included in the test report:

f) voltage tap distance L;

g) specimen dimensions and cross-sectional area A;

h) resistivity $\rho_1 = R_1 A L^{-1}$ and $\rho_2 = R_2 A L^{-1}$.

9.5 RRR value

The RRR value shall be quoted as $r_{\mathrm{RRR}} \pm u_{\mathrm{RRR}}$, for example 300 ± 15 ($k = 2$), where u_{RRR} is the combined standard uncertainty per Annex C. Alternately, r_{RRR} may be quoted as a minimum value, for example 285 minimum, to denote the lower limit of the confidence interval represented by the uncertainty. It is not necessary to report the uncertainty for a single measurement.

Additional information relating to the measurement of RRR is given in Annex A. Annex B describes definitions and an example of uncertainty in measurement. Uncertainty evaluation in the reference test method of RRR for composite superconductors is given in Annex C.

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Annex A
(Informative)

Additional information relating to the measurement of RRR

A.1 Considerations for specimens and apparatus

The requirements in Clause 4 imply several general guidelines for preparing specimens and the configuration of the measurement apparatus:

a) Niobium sheet stock is typically 2 mm to 5 mm thick. This implies a typical cross-sectional area A of a sample of ~10 mm^2 = 0,1 cm^2 if a bar is machined with width approximately the same as the sheet thickness.

b) Voltage tap separation depends on the apparatus dimensions, but cannot be longer than about 80 % of the length of the niobium bar cut from sheets. To conserve liquid helium, this is about 10 cm maximum, so a voltage tap separation L of 2 cm to 5 cm is reasonable.

c) Given that the resistivity ρ of pure niobium at 293 K is approximately 15 μΩ-cm, a typical resistance of the niobium bar is $R = \rho L/A$ = 15 μΩ-cm × 5 cm / 0,1 cm^2 = 750 μΩ.

d) If r_{RRR} = 300, then a resistance of 750 / 300 = 2,5 μΩ can be expected at ~10 K. Thus, given the scope described in Clause 1, a resistance of 1 μΩ to 20 μΩ should be expected.

e) To produce a measurement signal of ~ 1 μV at 10 K, as required by Clause 4, a current of 1 μV / 2,5 μΩ = 0,4 A will be required. Thus, a target of 1 A measuring current should be used to provide ample allowance for variations in RRR among different specimens. This corresponds to a current density of approximately 0,1 A/mm^2.

f) As an alternate guideline, assuming a voltage of 1 μV at 10 K is produced by 1 A measuring current for r_{RRR} = 300, then (L/A) = 1 μΩ × 300 / 15 μΩ-cm = 20 cm^{-1}. If the sample width w is the same as its 0,2 cm to 0,5 cm thickness, then the aspect ratio of the sample $L/w = (L/A) \times (A/w)$ = 20 cm^{-1} × (w^2/w) = 20 cm^{-1} × w is approximately 4 to 10. This justifies the requirement d) of Clause 4.

   With a measuring current of 1 A, a contact resistance of 1 mW / (1 A)^2 = 1 mΩ should be achieved. This resistance is typical of that produced by contacts with ~1 mm^2 area. Examples are

   1) a clean set screw contacting clean Nb metal,
   2) a clean conductive spring clip contacting clean Nb metal,
   3) a conductive terminal clamp anchored by a screw or spring, or
   4) a tightly wound fine copper wire (diameter about 0,2 mm) that surrounds the contact region, with solder connection between current leads and wire.

   Polishing the contact area, or applying a soft metal such as indium, may be used to reduce contact resistance. Contacts with small area, such as pin contacts or blade contacts, will probably not yield a suitable contact resistance.

g) To provide proper thermal sinking and thermal contact to thermometers, good thermal conductors should be used to support the sample. Such materials include copper, silver, or aluminum. Mild alloys of these metals increase the mechanical strength without greatly reducing the thermal conductivity.

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A.2 Considerations for specimen mounting orientation

The orientation of the specimen relative to the cryostat is not specified. The considerations in Clause A.1 result in a specimen that can be much longer than the typical width of the orifice at the top of a measurement dewar. Long samples are therefore conveniently inserted through a small dewar orifice in a vertical orientation. However, thermal gradients along the specimen may also result in such cases. Horizontal orientation may reduce significantly any thermal gradients, but might also place undue constraints on the sample dimensions. “U”-shaped specimens both accommodate the ability to lie flat in a horizontal orientation as well as provide a sufficient length between voltage taps, as long as no bending strain has been introduced during the specimen preparation procedure.

A.3 Alternative methods for increasing temperature of specimen above superconducting transition temperature

A.3.1 General

The following methods are also recommended for increasing temperature above the superconducting transition of the specimen. The rate of increasing temperature of the whole specimen within a range between 0,1 K/min and 10 K/min should be applied for these methods. In order to dampen the rate of increasing temperature and to avoid a large temperature gradient, special care should be taken in selecting heater power, heat capacity (the specimen with the measuring mandrel or the measuring base plate) and the distance between the heater and the specimen.

A.3.2 Heater method

The specimen can be heated above the superconducting transition by a heater installed in the measurement mandrel or in the measurement base plate after taking the specimen out of the liquid helium bath in the cryostat.

A.3.3 Controlled methods

a) Adiabatic method: In this method, the cryostat holds a chamber in which the specimen, a sample holder, a heater and so on are contained. Before the chamber is immersed in the liquid helium bath, air inside the chamber is pumped out and helium gas is filled. Then, the chamber is immersed in the liquid helium bath and the specimen is cooled to a temperature below the critical temperature. After the helium gas is pumped out, the specimen can be heated above the superconducting transition by the heater under adiabatic condition.

b) Quasi-adiabatic method: In this method, the cryostat holds the specimen a certain distance above the liquid helium bath for the entire cryogenic measurement. A thermal anchor from the measurement mandrel or the measurement base plate to the liquid helium bath allows the specimen to be cooled to a temperature below the critical temperature. The specimen can be heated above the superconducting transition by a heater located in the measurement mandrel or the measurement base plate under quasi-adiabatic condition.

c) Refrigerator method: In this method, an electromechanical apparatus (a refrigerator) is used to cool the specimen, which is mounted to a measurement mandrel or a measurement base plate, to a temperature below the critical temperature. The specimen can be heated above the superconducting transition by a heater or by controlling the refrigerator power.

A.4 Other test methods

A.4.1 General

A recent document [6] has compared several methods for determining the residual resistivity ratio of niobium.

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