SLOVENSKI STANDARD
oSIST prEN IEC 61788-23:2023

01-september-2023

Superprevodnost - 23. del: Meritve deleža preostale upornosti - Delež preostale upornosti niobijskih superprevodnikov

Superconductivity - Part 23: Residual resistance ratio measurement - Residual resistance ratio of cavity-grade Nb superconductors

Supraleitfähigkeit - Teil 23: Messung des Restwiderstandsverhältnisses - Restwiderstandsverhältnis von hochreinen Nb-Supraleitern für Kavitäten

Supraconductivité - Partie 23: Mesurage du rapport de résistance résiduelle - Rapport de résistance résiduelle des supraconducteurs de Nb à cavités

Ta slovenski standard je istoveten z: **prEN IEC 61788-23:2023**

ICS:

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29.050	Superprevodnost in prevodni materiali	Superconductivity and conducting materials

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90/503/CDV

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IEC TC 90 : SUPERCONDUCTIVITY	
SECRETARIAT: Japan	SECRETARY: Mr Jun Fujikami
OF INTEREST TO THE FOLLOWING COMMITTEES:	PROPOSED HORIZONTAL STANDARD: ☐ Other TC/SCs are requested to indicate their interest, if any, in this CDV to the secretary.
FUNCTIONS CONCERNED: ☐ EMC ☐ ENVIRONMENT ☐ QUALITY ASSURANCE ☐ SAFETY	
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TITLE:

Superconductivity - Part 23: Residual resistance ratio measurement - Residual resistance ratio of cavity-grade Nb superconductors

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NOTE FROM TC/SC OFFICERS:

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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SUPERCONDUCTIVITY –

Part 23: Residual resistance ratio measurement – Residual resistance ratio of cavity-grade Nb superconductors

FOREWORD

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IEC 61788-23 has been prepared by IEC technical committee 90: Superconductivity. It is an International Standard.

This second edition cancels and replaces the first edition published in 2018. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

a) The scope of this standard was modified to restrict the range of residual resistance ratio to that encountered by providers of material for superconducting radio-frequency cavities.

b) The references to technical material were updated and corrected.

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

FDIS	Report on voting
90/478/FDIS	90/482/RVD

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this International Standard is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/standardsdev/publications.

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

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

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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 RRR 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 millimetres 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 this 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. Note that systematic differences of up to 10 % are produced by these other methods, which is larger than the target uncertainty of this document. It is therefore important 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 Clause C.2.

SUPERCONDUCTIVITY –

Part 23: Residual resistance ratio measurement – Residual resistance ratio of cavity-grade 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 150 < r_{RRR} < 600. The test method is 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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1
residual resistance ratio
RRR
r_{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;

R_2 is the resistance just above the superconducting transition, at ~10 K.

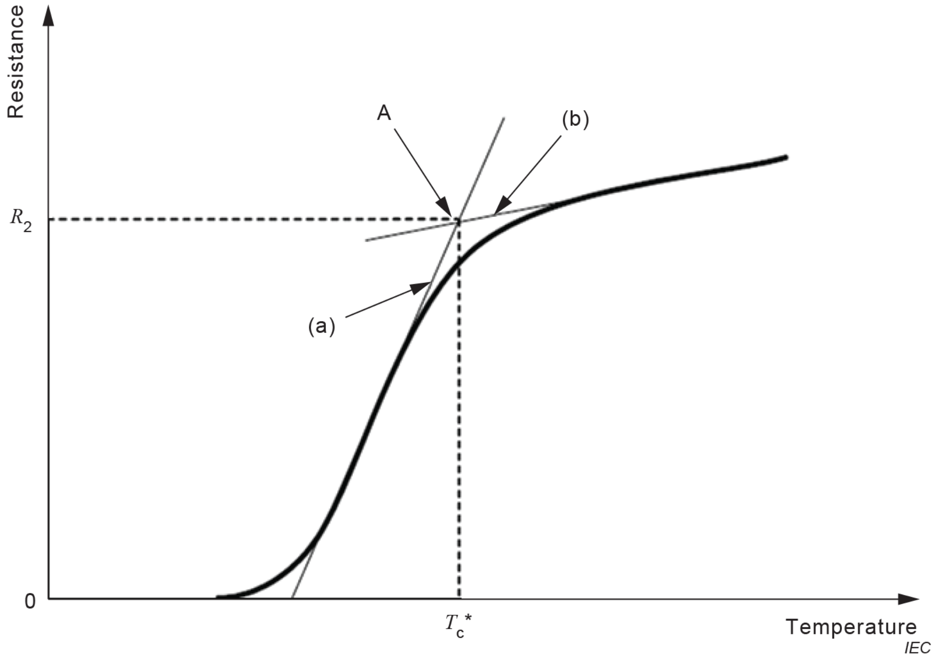

Figure 1 – Relationship between temperature and resistance near the superconducting transition

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 Figure 1, 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], [6], 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.

4 Principle

The 4-point DC electrical resistance technique shall be performed both at room temperature and at cryogenic temperature. The test of resistance shall be done as a function of temperature. Another test method of resistance as a function of time with increasing temperature is described in Annex A.4.2.

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.

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 are 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 is 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 aluminium, 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 aluminium alloys, and electrical grades of silver alloys. These provide high thermal conductivity and serve to remove thermal gradients during measurement. The specimen shall be insulated from the mandrel. Possible insulating materials include polyethylene terephthalate, polyester, and polytetrafluoroethylene, which may be applied as foils, tapes, or coatings. Glass-fibre 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 centre 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.

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 when 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. Alternatively, 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. The heater should be distributed along the mandrel and excessive power settings should be avoided. For instance, a point source of 1 W heat input operating at the centre of a 1 cm^2 mandrel upon which a 5 cm sample is mounted could produce temperature difference 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 can 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 shall be protected and immobilized against a support piece during transport. Operations to de-burr samples shall 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 that coating the entire specimen is avoided.

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. Installation and instrumentation of the specimen shall not apply excessive force, bending strain, tensile strain, or torsion to the specimen.