SLOVENSKI STANDARD
oSIST prEN 61788-23:2017

01-september-2017

Superprevodnost - 23. del: Meritve razmerja preostale upornosti - Razmerje preostale upornosti superprevodnikov iz niobija (Nb)

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

Ta slovenski standard je istoveten z: **prEN 61788-23:2017**

ICS:

29.050	Superprevodnost in prevodni materiali	Superconductivity and conducting materials

oSIST prEN 61788-23:2017 **en**

90/387/CDV

COMMITTEE DRAFT FOR VOTE (CDV)

PROJECT NUMBER: **IEC 61788-23 ED1**	
DATE OF CIRCULATION: **2017-05-26**	CLOSING DATE FOR VOTING: **2017-08-18**
SUPERSEDES DOCUMENTS: **90/380/CD,90/385/CC**	

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

☒ SUBMITTED FOR CENELEC PARALLEL VOTING

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Attention IEC-CENELEC parallel voting

The attention of IEC National Committees, members of CENELEC, is drawn to the fact that this Committee Draft for Vote (CDV) is submitted for parallel voting.

The CENELEC members are invited to vote through the CENELEC online voting system.

This document is still under study and subject to change. It should not be used for reference purposes.

Recipients of this document are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

TITLE:

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

NOTE FROM TC/SC OFFICERS:

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

SUPERCONDUCTIVITY –

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

FOREWORD

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

This standard deals with the test method of RRR of high-purity Nb superconductor used to make superconducting radio-frequency cavities, composite superconducting wires, and other components where Nb purity is essential.

The text of this standard is based on the following documents:

FDIS	Report on voting
90/XX/FDIS	90/XX/RVD

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

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

A list of all parts of 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 publication will remain unchanged until the stability date[1)] indicated on the IEC web site under "http://webstore.iec.ch" in the data related to the specific publication. At this date, the publication will be

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

1) The National Committees are requested to note that for this publication the stability date is 2023

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 International Standard 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. 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 International Standard from the 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 for the low resistance value. A comparison between the standard 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 standard. Care should therefore be taken to apply the standard 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 also described in Annex C.

SUPERCONDUCTIVITY –

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

1 Scope

The following document 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 References

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

(2) IEC 61788-4 Ed.4 Superconductivity – Part 4: Residual resistance ratio measurement –Residual resistance ratio of Nb-Ti and Nb_3Sn composite superconductors

(3) IEC 61788-10 Ed.2 Superconductivity – Part 10: Critical temperature measurement - Critical temperature of composite superconductors by a resistance method

(4) ASTM B393-09e1, Standard Specification for Niobium and Niobium Alloy Strip, Sheet, and Plate, ASTM International, West Conshohocken, PA, 2009, www.astm.org

(5) GOODRICH L. F, STAUFFER T. C., SPLETT J. D., and VECCHIA D. F., Measuring Residual Resistivity Ratio of High-Purity Nb, NIST publication 31484, also published in Advances in Cryogenic Engineering (Materials) 50A, 41 (2004)

(6) SINGER W., ERMAKOV A., and SINGER X., RRR-measurement techniques on high purity niobium Tesla Technology Collaboration report 2010-02 (2010) at http://flash.desy.de/reports_publications/tesla_reports/ttc_reports_2010/.

3 Terms and definitions

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

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

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

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

Note 1 to entry: In this standard, 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

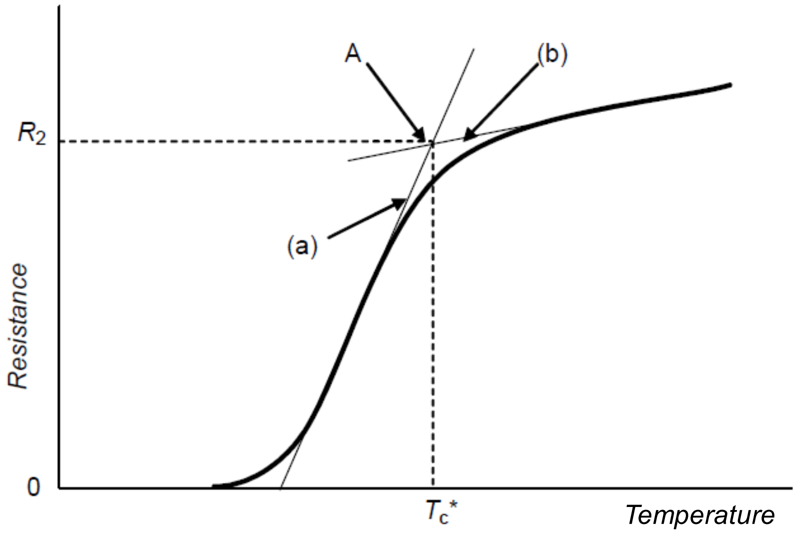

Figure 1 – Relationship between temperature and resistance near the superconducting transition.

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 (2), and should not be confused with the value defined at the midpoint of the transition, called T_c^* in (3).

Note 3 to entry: Some standards or documented techniques, e.g. (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 International Standard 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 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 order 1 μV. For electrical safety, maximum current density should never exceed 1 A mm^{-2}.

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 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. Glass-fiber reinforced epoxy or other composite materials with good thermal conductivity at cryogenic temperature may 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.

If the mandrel or base plate is metal, then the surface of the mandrel or base plate intended to lie against the niobium specimen shall be covered with an electrically insulating material. Possible insulating materials include polyethylene terephthalate, polyester, and polytetrafluoroethylene, which may be applied as foils, tapes, coatings, etc.

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 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.

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 to prevent excessive boil-off due to heat conduction from the surrounding laboratory, such as thin-walled stainless steel tubes, composite