IEC 61788-4

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INTERNATIONAL STANDARD

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Superconductivity –
Part 4: Residual resistance ratio measurement – Residual resistance ratio of Nb-Ti and Nb_3Sn composite superconductors

Supraconductivité –
Partie 4: Mesurage du rapport de résistance résiduelle – Rapport de résistance résiduelle des composites supraconducteurs de Nb-Ti et de Nb_3Sn

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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 4: Residual resistance ratio measurement – Residual resistance ratio of Nb-Ti and Nb_3Sn composite superconductors

FOREWORD

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International Standard IEC 61788-4 has been prepared by IEC technical committee 90: Superconductivity.

This fifth edition cancels and replaces the fourth edition published in 2016. This edition constitutes a technical revision.

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

a) change in the suitable distance of voltage taps on the specimen for reliable measurement,

b) new report on the result of the round robin test of the residual resistance ratio of Nb_3Sn superconductors that proves the validity of the measurement method in this standard,

c) revision of the confusing definitions of the copper ratio and copper fraction.

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

FDIS	Report on voting
90/448/FDIS	90/451/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 indicated on the IEC website 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- withdrawn,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.

IMPORTANT – The 'colour inside' logo on the cover page of this publication indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

INTRODUCTION

Copper, Cu/Cu-Ni or aluminium is used as matrix material in Ni-Ti and Nb_3Sn composite superconductors and works as an electrical shunt when the superconductivity is interrupted. It also contributes to recovery of the superconductivity by conducting heat generated in the superconductor to the surrounding coolant. The cryogenic-temperature resistivity of copper is an important quantity, which influences the stability and AC losses of the superconductor. The residual resistance ratio is defined as a ratio of the resistance of the superconductor at room temperature to that just above the superconducting transition.

This document specifies the test method for residual resistance ratio of Nb-Ti and Nb_3Sn composite superconductors. The curve method is employed for the measurement of the resistance just above the superconducting transition. Other methods are described in Clause A.3.

SUPERCONDUCTIVITY –

Part 4: Residual resistance ratio measurement – Residual resistance ratio of Nb-Ti and Nb_3Sn composite superconductors

1 Scope

This part of IEC 61788 specifies a test method for the determination of the residual resistance ratio (RRR) of Nb-Ti and Nb_3Sn composite superconductors with Cu, Cu-Ni, Cu/Cu-Ni and Al matrix in a strain-free condition and zero external magnetic field. This method is intended for use with superconductor specimens that have a monolithic structure with rectangular or round cross-section, RRR value less than 350, and cross-sectional area less than 3 mm^2. In the case of Nb_3Sn, the specimens have received a reaction heat-treatment.

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 (IEV) – 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
ratio of resistance at room temperature to the resistance just above the superconducting transition

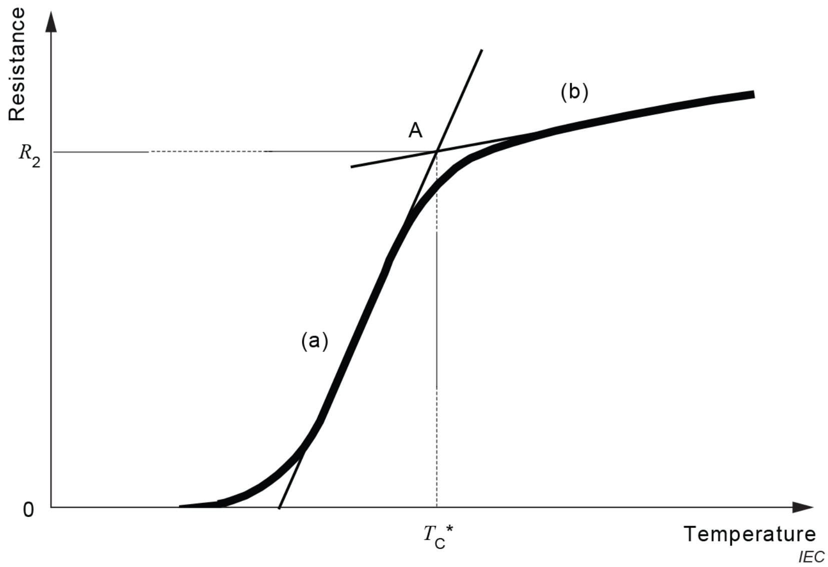
Note 1 to entry: This note applies to the French language only.

Note 2 to entry: In this document for Nb-Ti and Nb_3Sn composite superconductors, the room temperature is defined as 293 K (20 °C), and the residual resistance ratio is obtained in Formula (1), where the resistance (R_1) at 293 K is divided by the resistance (R_2) just above the superconducting transition.

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

Here r_{RRR} is a value of the residual resistance ratio, R_2 is a value of the resistance measured in a strain-free condition and zero external magnetic field.

Figure 1 shows schematically a resistance versus temperature curve acquired on a specimen while measuring the cryogenic resistance.–



The cryogenic resistance, R_2, is determined by the intersection, A, of two straight lines (a) and (b) at temperature T_c*.

Figure 1 – Relationship between temperature and resistance

4 Principle

The resistance measurement both at room and cryogenic temperatures shall be performed with the four-terminal technique. All measurements are done without an applied magnetic field.

The target relative combined standard uncertainty of this method is defined as an expanded uncertainty ($k = 2$) not to exceed 5 %.

The maximum bending strain induced during mounting and cooling the Nb-Ti specimen shall not exceed 2 %. The measurement shall be conducted in a strain-free condition or in a condition with allowable thermal strain for the Nb_3Sn specimen.

5 Apparatus

5.1 Material of measurement mandrel or of measurement base plate

Material of the measurement mandrel for a coiled Nb-Ti specimen or of the measurement base plate for a straight Nb-Ti or Nb_3Sn specimen shall be copper, aluminium, silver, or the like whose thermal conductivity is equal to or better than 100 W/(m·K) at liquid helium temperature (4,2 K). The surface of the material shall be covered with an insulating layer (tape or a layer made of polyethylene terephthalate, polyester, polytetrafluoroethylene, etc.) whose thickness is 0,1 mm or less.

5.2 Diameter of the measurement mandrel and length of the measurement base plate

The diameter of the measurement mandrel shall be large enough to keep the bending strain of the specimen less than or equal to 2 % for the Nb-Ti specimen. The Nb_3Sn specimen on a base plate shall be measured in a strain-free condition or a condition with allowable thermal strain.

The measurement base plate shall be at least 30 mm long in one dimension.

5.3 Cryostat for the resistance (R_2) measurement

The cryostat shall include a specimen support structure and a liquid helium reservoir for measurement of the resistance R_2. The specimen support structure shall allow the specimen, which is mounted on a measurement mandrel or a measurement base plate, to be lowered into and raised out of a liquid helium bath. In addition, the specimen support structure shall be made so that a current can flow through the specimen and the resulting voltage generated along the specimen can be measured.

6 Specimen preparation

The test specimen shall have no joints or splices with a length of 30 mm or longer. The specimen shall be instrumented with current contacts near each of its ends and a pair of voltage contacts over its central portion. The distance between two voltage taps (L) shall be 15 mm or longer. A thermometer for measuring cryogenic temperature shall be attached near the specimen.

Some mechanical method shall be used to hold the specimen against the insulated layer of the measurement mandrel or base plate. Special care should be taken during instrumentation and installation of the specimen on the measurement mandrel or base plate so that no excessive force, which may cause undesired bending strain or tensile strain, would be applied to the specimen. Ideally, the Nb_3Sn specimen is intended to be as straight as possible; however, this is not always the case, thus care should be taken to measure the specimen in its as received condition.

The specimen shall be mounted on a measurement mandrel or on a measurement base plate for these measurements. Both resistance measurements, R_1 and R_2, shall be made on the same specimen and the same mounting.

7 Data acquisition and analysis

7.1 Resistance (R_1) at room temperature

The mounted specimen shall be measured at room temperature (T_m (K)), where T_m satisfies the following condition: 273 K ≤ T_m ≤ 308 K. A specimen current (I_1(A)) shall be applied so that the current density is in the range of 0,1 A/mm^2 to 2 A/mm^2 based on the total wire cross-sectional area, and the resulting voltage (U_1 (V)), I_1 and T_m shall be recorded. Formula (2) below shall be used to calculate the resistance (R_m) at room temperature. The resistance (R_1) at 293 K (20 °C) shall be calculated using Formula (3) for a wire with Cu matrix. For wires that do not contain a pure Cu component, the value of R_1 shall be set equal to R_m, without any temperature correction.

$$R_m = \frac{U_1}{I_1} \tag{2}$$

$$R_1 = \frac{R_m}{\left[1+0{,}00393\times\left(T_m-293\right)\right]} \tag{3}$$

7.2 Resistance (R_2 or R_2^*) just above the superconducting transition

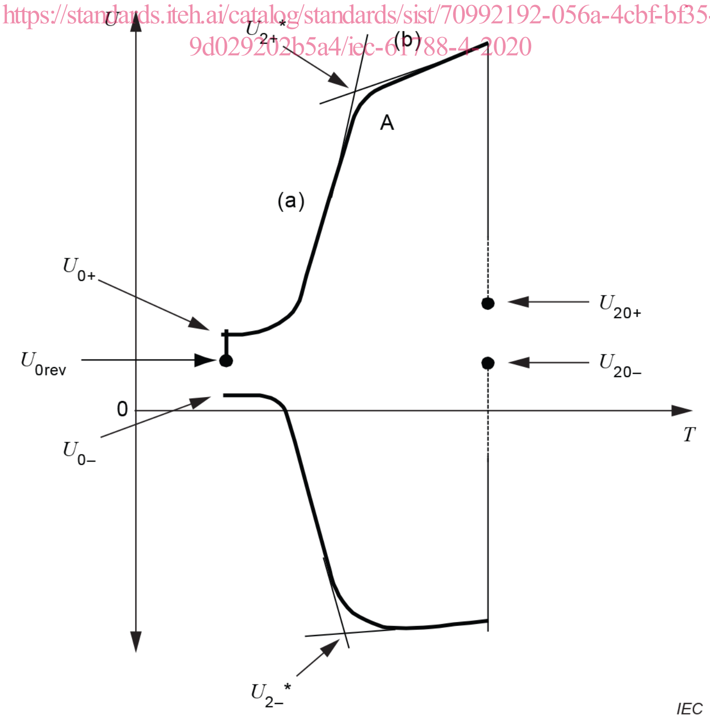
7.2.1 Correction of strain effect

Under a strained condition of the Nb-Ti specimen, the measured cryogenic resistance, R_2^*, is not a correct value for R_2. The corresponding correction of the strain effect is described in 7.3.

7.2.2 Data acquisition of cryogenic resistance

The specimen, which is still mounted as it was for the room temperature measurement, shall be placed in the cryostat for electrical measurement specified in 5.3. Horizontal mounting of the specimen is recommended in Clause A.1. Alternative cryostats that employ a heating element to sweep the specimen temperature are described in Clause A.2. The specimen shall be slowly lowered into the liquid helium bath and cooled to liquid helium temperature over a time period of at least 5 min.

During the acquisition phases of the low-temperature R_2^* measurements, a specimen current (I_2) shall be applied so that the current density is in the range 0,1 A/mm^2 to 10 A/mm^2 based on the total wire cross-sectional area, and the resulting voltage (U (V)), I_2 (A), and specimen temperature (T (K)) shall be recorded. In order to keep the ratio of signal to noise high enough, the measurement shall be carried out under the condition that the absolute value of the resulting voltage above the superconducting transition exceeds 10 µV. An illustration of the data to be acquired and its analysis is shown in Figure 2.



NOTE Voltages with subscripts + and – are those obtained in the first and second measurements under positive and negative currents, respectively, and U_{20+} and U_{20-} are those obtained at zero current. For clarity, U_{0rev}, measured at zero current is not shown coincident with U_{0-}. Straight line (a) is drawn in the transition region with a sharp increase in the voltage with temperature and straight line (b) is drawn in the region with a nearly constant voltage.

Figure 2 – Voltage versus temperature curves and definitions of each voltage

When the specimen is in the superconducting state and the test current (I_2) is applied, two voltages shall be measured nearly simultaneously: U_{0+} (the initial voltage recorded with a positive current polarity) and $U_{0\text{rev}}$ (the voltage recorded during a brief change in applied current polarity). A valid R_2^* measurement requires that excessive interfering voltages are not present and that the specimen is initially in the superconducting state. Thus, the following formulae shall be met for a valid measurement:

$$\frac{\left|U_{0+} - U_{0\text{rev}}\right|}{\overline{U}_2} < 1\ \% \tag{4}$$

where $\overline{U}_2$ is the average voltage for the specimen in the normal state at cryogenic temperature, which is defined by Formula (5).

The specimen shall be gradually warmed so that it changes to the normal state completely. When the cryostat for the resistance measurement specified in 5.3 is used, this can be achieved simply by raising the specimen to an appropriate position above the liquid helium level. The specimen voltage versus temperature curve shall be acquired with the rate of temperature increase maintained between 0,1 K/min and 10 K/min. The voltage versus temperature curve shall continue to be recorded during the transition into the normal state, up to a temperature somewhat less than 15 K for the Nb-Ti specimen and less than 25 K for the Nb_3Sn specimen. Then, the specimen current shall be decreased to zero and the corresponding voltage, U_{20+}, shall be recorded at a temperature below 15 K for the Nb-Ti specimen and below 25 K for the Nb_3Sn specimen.

The specimen shall then be slowly lowered into the liquid helium bath and cooled to within ±1 K from the temperature at which the initial voltage signal U_{0+} was recorded. A specimen current, I_2, with the same magnitude but negative polarity (polarity opposite that used for the initial curve) shall be applied and the voltage U_{0-} shall be recorded at this temperature. The procedural steps shall be repeated to record the voltage versus temperature curve with this negative current. In addition, when the measurement current, I_2, decreases to 0, the recording of U_{20-} shall be made at within ±1 K from the temperature at which U_{20+} was recorded.

Each of the two voltage versus temperature curves shall be analysed by drawing a line (a) through the data where the absolute value of voltage sharply increases with temperature (see Figure 2) and drawing a second line (b) through the data above the transition where the voltage is nearly constant for Nb-Ti or raised gradually and almost linearly for Nb_3Sn with temperature increase. U_{2+}^* and U_{2-}^* in Figure 2 shall be determined at the intersection of these two lines for the positive and negative polarity curves, respectively.

The corrected voltages, U_{2+} and U_{2-}, shall be calculated using the following equations: $U_{2+} = U_{2+}^* - U_{0+}$ and $U_{2-} = U_{2-}^* - U_{0-}$. The average voltage, $\overline{U}_2$, shall be defined as

$$\overline{U}_2 = \frac{\left|U_{2+} - U_{2-}\right|}{2} \tag{5}$$

A valid R_2^* measurement requires that the shift of thermoelectric voltage be within acceptable limits during the measurements of U_{2+} and U_{2-}. Thus, the following condition shall be met for a valid measurement:

$$\frac{|\Delta_+ - \Delta_-|}{\bar{U}_2} < 3\ \% \tag{6}$$

where Δ_+ and Δ_- are defined as $\Delta_+ = U_{20+} - U_{0+}$ and $\Delta_- = U_{20-} - U_{0-}$. If the R_2^* measurement does not meet the validity requirements in 7.2.2, specifically either in Formula (4) or (6), then improvement steps either in hardware or experimental operation shall be taken to meet these requirements before results are reported.

Formula (7) shall be used to calculate the measured resistance (R_2^*) just above the superconducting transition.

$$R_2^* = \frac{\bar{U}_2}{I_2} \tag{7}$$

7.2.3 Optional acquisition methods

The method described in the main clauses of this document is the “reference” method and optional acquisition methods are outlined in Clause A.3.

7.3 Correction on measured R_2^* of Nb-Ti composite superconductor for bending strain

If there is no pure Cu component in the superconductor, then R_2 shall be set equal to R_2^*.

For a specimen with a pure Cu component, the bending strain shall be defined by ε_b = 100 × (h/r) (%), where h is a half of the specimen thickness for rectangular wires or a radius for round wires and r is the bending radius. If the bending strain is less than 0,3 %, then no correction is necessary, and R_2 shall be set equal to R_2^*.

If neither of the above two situations applies, then the resistance R_2 just above the superconducting transition under the strain-free condition shall be estimated by

$$R_2 = R_2^* - \Delta\rho \times \frac{L}{S_{Cu}} \tag{8}$$

where $\Delta\rho$ is defined below and S_{Cu} and L are defined in 8.4. The increase in the resistivity of pure copper at 4,2 K due to tensile strain, ε (%), is expressed by

$$\Delta\rho\ (\Omega\text{m}) = 6{,}24 \times 10^{-12}\varepsilon - 5{,}11 \times 10^{-14}\varepsilon^2;\ \varepsilon \le 2\ \% \tag{9}$$

The calculation of Formula (9) shall be carried out assuming that the equivalent tensile strain ε is (1/2)ε_b and (4/3 π)ε_b for rectangular and round wires, respectively. The bending strain dependency of residual resistance ratio for pure copper is described in Clause A.4.

7.4 Residual resistance ratio (RRR)

The RRR value shall be calculated using Formula (1).

8 Uncertainty and stability of the test method

8.1 Temperature

The room temperature shall be determined with a standard uncertainty not exceeding 0,6 K, while holding the specimen, which is mounted on the measurement mandrel or on the measurement base plate, at room temperature.

8.2 Voltage

For the resistance measurement, the voltage signal shall be measured with a relative standard uncertainty not exceeding 0,3 %.

8.3 Current

When the current is directly applied to the specimen with a programmable DC current source, the specimen test current shall be determined with a relative standard uncertainty not exceeding 0,3 %.

When the specimen test current is determined from a voltage-current characteristic of a standard resistor by the four-terminal technique, the standard resistor, with a relative combined standard uncertainty not exceeding 0,3 %, shall be used.

The fluctuation of DC specimen test current, provided by a DC power supply, shall be less than 0,5 % during every resistance measurement.

8.4 Dimension

The distance along the specimen between the two voltage taps (L) shall be determined with a relative combined standard uncertainty not exceeding 5 %.

For correction of the bending strain effect in the case of the wire with pure Cu matrix, the cross-sectional area of Cu matrix (S_{Cu}) shall be determined using a nominal value of copper to non-copper ratio and nominal dimensions of the specimen. The wire diameter (d) and mandrel radius (R_{d}) shall be determined with relative standard uncertainty not exceeding 1 % and 3 %, respectively.

9 Test report

9.1 RRR value

The obtained RRR value (r_{RRR}) shall be reported as

$$r_{\mathrm{RRR}}\left(1 \pm U_{\mathrm{re}}\right) \quad (n = \cdots), \tag{10}$$

where

U_{re} is the expanded relative uncertainty:

$$U_{\mathrm{re}} = 2u_{\mathrm{r}} \quad (k = 2)$$

where

u_{r} denotes the relative combined standard uncertainty,

k is a coverage factor, and