IEC 61788-5

Edition 2.0 2013-05

INTERNATIONAL STANDARD

NORME INTERNATIONALE

Superconductivity – Part 5: Matrix to superconductor volume ratio measurement – Copper to superconductor volume ratio of Cu/Nb-Ti composite superconducting wires

Supraconductivité – Partie 5: Mesure du rapport volumique matrice/supraconducteur – Rapport volumique cuivre/supraconducteur des fils en composite supraconducteur Cu/Nb-Ti

IEC 61788-5:2013

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INTERNATIONAL
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INTERNATIONALE

PRICE CODE
CODE PRIX T

ICS 17.220.20; 29.050 ISBN 978-2-83220-831-1

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FOREWORD

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International Standard IEC 61788-5 has been prepared by IEC technical committee 90: Superconductivity.

This second edition cancels and replaces the first edition published in 2000. It constitutes a technical revision. The main revisions are the addition of two new annexes, "Uncertainty considerations" (Annex E) and "Uncertainty evaluation in test method of copper to superconductor volume ratio of Cu/Nb-Ti composite superconductors" (Annex F).

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

FDIS	Report on voting
90/321/FDIS	90/324/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 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- withdrawn,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.

INTRODUCTION

The copper to superconductor volume ratio of composite superconductors is used mainly to calculate the critical current density of superconducting wires. The test with the method given in this International Standard may be used to provide part of the information needed to determine the suitability of a specific superconductor. Moreover, this method is useful for quality control, acceptance or research testing if the precautions given in this standard are observed.

The test method given in this International Standard is based on the condition that the specific mass of Nb-Ti is known or the Nb-Ti alloy fraction is known and Annex B can be used to estimate the specific mass. If the specific mass of Nb-Ti is unknown and the Nb-Ti alloy fraction is unknown and/or the fraction of Nb barrier is unknown, another method to determine the copper to superconductor volume ratio of composite superconductors is described in Annex A.

SUPERCONDUCTIVITY –

Part 5: Matrix to superconductor volume ratio measurement – Copper to superconductor volume ratio of Cu/Nb-Ti composite superconducting wires

1 Scope

This part of IEC 61788 covers a test method for the determination of copper to superconductor volume ratio of Cu/Nb-Ti composite superconducting wires.

This test method and the alternate method in Annex A are intended for use with Cu/Nb-Ti composite superconducting wires with a cross-sectional area of 0,1 mm^2 to 3 mm^2, a diameter of the Nb-Ti filament(s) of 2 μm to 200 μm, and a copper to superconductor volume ratio of 0,5 or more.

The Cu/Nb-Ti composite test conductor discussed in this method has a monolithic structure with a round or rectangular cross-section. This test method is carried out by dissolving the copper with nitric acid. Deviations from this test method that are allowed for routine tests and other specific restrictions are given in this standard.

Cu/Nb-Ti composite superconducting wires beyond the limits in the cross-sectional area, the filament diameter and the copper to superconductor volume ratio could be measured with this present method with an anticipated reduction of uncertainty. Other, more specialized, specimen test geometries may be more appropriate for conductors beyond the limits and have been omitted from this present standard for simplicity and to retain low uncertainty.

The test method given in this standard is expected to apply to other superconducting composite wires after some appropriate modifications.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 (all parts), *International Electrotechnical Vocabulary* (available at <http://www.electropedia.org>)

3 Terms and definitions

For the purposes of this document, the definitions given in IEC 60050-815 as well as the following definition apply.

3.1
copper to superconductor volume ratio
ratio of the volume of the copper stabilizing material to the volume without copper consisting of Nb-Ti filaments and their Nb barriers

4 Principle

The test method utilizes the nature of the Cu/Nb-Ti composite superconducting wire whereby the copper dissolves in nitric acid solution but the Nb-Ti filaments and Nb barriers do not.

After measuring its mass, dip the specimen into the nitric acid solution to dissolve only the copper.

Then measure the mass of the remaining Nb-Ti filaments and their Nb barriers.

The volume and mass of the starting wire and the mass of the filaments are used to determine the copper to superconductor volume ratio.

5 Chemicals

The following chemicals shall be prepared for sample preparation:

a) nitric acid solution consisting of nitric acid (a volume fraction of 50 % to 65 % recommended) and distilled water;
b) organic solvent;
c) degreasing solvent;
d) ethyl alcohol;
e) distilled (pure) water.

NOTE When nitric acid of more than a mass fraction of 65 % is used, the acid is diluted with distilled water within the range of the above content.

6 Apparatus

The following apparatus shall be prepared.

- Draft chamber
- Balance

A balance shall have a manufacturer's specified uncertainty of ±0,1 mg or better.

- Dryer or drying oven

A dryer or a drying oven shall be used for evaporating moisture after washing the specimen.

- Beaker
- Watch-glass
- Plastic tweezers
- Filter papers
- Thermometer
- Rubber gloves and protection spectacles

Rubber gloves and protection spectacles shall be used for protecting the human body from the harmful acid liquid or fumes. The dissolution of the specimen shall be performed in a draft chamber in order to protect the human body.

7 Measurement procedure

7.1 Quantity of specimen

Take a specimen of around 1 g to 10 g in mass from the base test material.

7.2 Removal of insulating cover material

An appropriate organic solvent, which does not erode the copper, shall be used to remove any insulating cover material of the specimen. Finally, it shall be visually checked that the insulating cover material no longer remains.

If no organic solvents can remove the insulating cover material, the mechanical removal in Annex C is an alternative.

7.3 Cleaning

A degreaser shall be used to remove oil and/or grease traces from the specimen, whose cover material has been removed. It shall then be cleaned with pure water. Finally, the degreased specimen shall be dipped into ethyl alcohol to replace the water. Cleaning without using ethyl alcohol is an alternative, by using the drying process described in 7.4.

7.4 Drying

The clean specimen shall be placed on a watch-glass and dried fully in a dryer or a drying oven at a temperature of 60 °C or lower with the holding time more than 0,5 hours. When cleaning the specimen is carried out without ethyl alcohol, the specimen shall be dried fully in a dryer or a drying oven at a temperature of 100 °C with the holding time more than 0,5 hours.

7.5 Measurement of specimen mass and its repetition

When the specimen is cooled down to 35 °C or lower, its mass shall be measured on a sheet of weighing paper, using a balance with a manufacturer's specified uncertainty of ±0,1 mg or better.

After completion of this mass measurement (the first measurement), remove the specimen from the balance.

To assure that the specimen has been fully dried, the mass of the specimen shall be measured again about 10 min after the first measurement (the second measurement).

The difference in mass between the first and second measurements shall be within ±0,5 %. If this difference is within ±0,5 %, the average of the two measurements shall be regarded as the mass of the specimen.

If the difference in mass is more than ±0,5 %, cleaning of the specimen with ethyl alcohol and drying of the specimen shall be repeated as described in 7.3, 7.4 and 7.5 until the difference in mass of the two measurements is within ±0,5 %.

As soon as this part of the method is qualified by a successful repetition, the second mass measurement can be omitted in subsequent measurements. However, periodic re-qualification shall be performed every six months or after changes of equipment or personnel.

7.6 Dissolving copper

The copper shall be dissolved from the specimen in the following manner.

Put approximately 150 ml of the nitric acid solution in a 300 ml beaker. Tie a knot in the specimen to help retain all of the filaments upon completion of the etch. In the draft chamber, while maintaining the temperature of the nitric acid solution between 20 °C and 50 °C, the whole specimen shall be dipped into the nitric acid solution for 30 min to 1 h to completely dissolve the copper of the specimen. It shall be checked visually that the copper has been completely dissolved. For wires with filaments less than 10 μm, the second etch according to Annex D is recommended to assure a complete copper dissolution.

Note that a fresh nitric acid solution shall be used for each specimen that is etched.

When copper is dissolved in the nitric acid solution, nitrite gas is generated. Because the nitric acid and the nitrite gas are harmful to the human body, use all safety precautions in handling acids such as wearing protective clothing and carrying out work to dissolve the copper in the draft chamber. In addition, the fumes generated during storage and use are harmful. Normal safety precautions for acid storage, use and disposal shall be followed.

Use rubber gloves, protection spectacles and a pair of plastic tweezers during the treatment of the nitric acid solution.

NOTE The temperature of the nitric acid solution specified here is that before dipping the specimen in it. The temperature can rise to more than 50 °C when dissolution of the copper is in progress.

When mixing the solution, always add the nitric acid to the water.

7.7 Cleaning and drying the Nb-Ti filaments

Cleaning and drying the Nb-Ti filaments shall be performed in the following manner.

Acid shall be carefully poured out of the beaker into a plastic sewage reservoir, keeping the specimen in the beaker so as not to lose any broken filaments. The beaker shall be refilled with distilled water to rinse. The water shall be carefully poured out of the beaker. The beaker shall now be refilled, with ethyl alcohol this time to replace any remaining water. Now to dry all of the filaments fully, the specimen shall be placed, using plastic tweezers, on a sheet of filter paper with any broken or loose filaments. They shall then be placed in a dryer or a drying oven (see 7.4).

If a green stain is noticed on the filter paper, then there is acid remaining on the filaments. This acid shall be removed by rinsing again in alcohol.

Cleaning without using ethyl alcohol is an alternative, by using the drying process described in 7.4.

If there are too many broken filaments, the procedures shall be repeated from the beginning on a new specimen.

Nb-Ti filaments with a diameter of about 10 μm or less can be combustible when they are removed from the acid and exposed to air after the matrix has been removed. Ignition sources (including flame, heat, spark and electrostatic discharge) are avoided. In addition, tweezers shall be used to handle the etched filaments and they shall not be put in contact with any part of the body. Normal safety precautions for metal combustion hazards shall be followed.

7.8 Measurement of dissolved specimen mass and its repetition

When the specimen is cooled down to 35 °C or lower, using a balance with a manufacturer's specified uncertainty of ±0,1 mg or better, the specimen shall be weighed as in 7.5. A sheet of weighing paper shall be used for the measurement to avoid losing broken filaments (the first measurement).

After completion of the mass measurement described in 7.5, the Nb-Ti filaments shall be removed from the balance. To know whether the Nb-Ti filaments have been fully dried, the mass of the Nb-Ti filaments shall be weighed again about 10 min after the first measurement (the second measurement).

The difference in mass shall be within ±0,5 % between the second measurement and the first measurement. If the difference in mass is within ±0,5 % between the two measurements, the average of the masses of the two measurements shall be regarded as the mass of the filaments.

If the difference in mass of the two measurements is more than ±0,5 %, only cleaning with ethyl alcohol and drying shall be repeated as described in the procedural step of 7.7, and then procedural steps shall be repeated again in the procedural step of 7.5. Then, check again to make sure that the difference in mass of the two measurements is within ±0,5 %.

As soon as this part of the method is qualified by a successful repetition, the second mass measurement can be omitted in subsequent measurements. However, periodic re-qualification shall be performed every six months or after changes in equipment or personnel.

7.9 Procedural repetition for second specimen

The procedural steps in 7.1 through 7.8 shall be repeated on the second specimen.

As soon as the method is qualified by a successful repetition, the repeated measurements on the second specimen can be omitted in subsequent measurements. However, periodic re-qualification shall be performed every six months or after changes in equipment or personnel.

8 Calculation of results

For each measurement, the copper to superconductor volume ratio shall be obtained down to two decimal places in the following equation, by rounding off to two decimal places.

If two specimens are measured, the average of the two ratios shall be regarded as the copper to superconductor volume ratio.

Copper to superconductor volume ratio is expressed as $\frac{(M_W - M_{Nb-Ti}) \times \rho_{Nb-Ti}}{M_{Nb-Ti} \times \rho_{Cu}}$,

where

M_W is the mass of the specimen g;

$M_{Nb\text{-}Ti}$ is the mass of the Nb-Ti filaments g;

ρ_{Cu} is 8,93, which is the specific mass of copper g/cm^3;

$\rho_{Nb\text{-}Ti}$ is the specific mass of the Nb-Ti filament g/cm^3.

The specific mass of the Nb-Ti alloy can be obtained by interpolation of the values given in Annex B if it is not given by the wire manufacturer.

NOTE If a barrier such as Nb is used, it is included in the mass of the Nb-Ti filament by calculating an effective filament specific mass taking into consideration the fraction of Nb barrier.

9 Uncertainty of the test method

The advantage of the method is that the copper to superconductor volume ratio can be obtained only from the masses of the specimen and Nb-Ti filaments. Since masses can be

measured fairly accurately, the masses can be determined with a relative combined standard uncertainty of less than 0,05 % even for a specimen with a mass of 1 g and a copper to superconductor volume ratio of 10.

Uncertainty is also affected by the specific mass of Nb-Ti. The first option shall be to use the value of the specific mass of Nb-Ti given by the wire manufacturer because it depends on more than the alloy composition (see NOTE 1 in Annex B).Otherwise, the value of the specific mass of Nb-Ti alloy shall be determined within a relative standard uncertainty of 0,5 % by interpolation of the values listed in Annex B.

If a barrier such as Nb is used, it shall be included in the mass of the Nb-Ti filament by calculating an effective filament specific mass taking into consideration the fraction of Nb barrier to retain low uncertainty.

If the specific mass of Nb-Ti is unknown and the Nb-Ti alloy fraction is unknown and/or the fraction of Nb barrier is unknown, then use the method in Annex A.

The target relative combined standard uncertainty of this test method shall not exceed 2 % (using a coverage factor of $k = 1$), which is confirmed in the relative combined standard uncertainty of 0,06 % for the copper dissolving method, and 0,2 % for the copper mass method according to round robin tests made to establish this standard as shown in Annex F.

10 Test report

10.1 Identification of test specimen

The test specimen shall be identified, if possible, by the following information:

a) manufacturer name of the specimen;
b) identification number;
c) billet number;
d) raw material composition;
e) shape and area of the cross-section of the wire, number of filaments, diameter of filaments, and Nb barrier.

10.2 Report of copper to superconductor volume ratio

The test report shall contain the following information:

a) the copper to superconductor volume ratio of each specimen;
b) Nb-Ti specific mass value used;
c) method of removing insulation from the specimen, if any.

10.3 Report of test conditions

The following test conditions shall be reported:

a) ambient temperature;
b) nitric acid temperature at the beginning;
c) nitric acid immersion time duration;
d) drying time duration.

Annex A
(normative)

Copper to superconductor volume ratio – copper mass method

A.1 General

If the specific mass of Nb-Ti is unknown and the Nb-Ti alloy fraction is unknown and/or the fraction of Nb barrier is unknown, then the copper to superconductor volume ratio shall be measured in the following manner. Clauses 1 to 6 also apply to this annex.

A.2 Quantity of specimen

A specimen of around 50 cm long and not exceeding the mass of 10 g shall be taken out of the base test material.

A.3 Remove insulation, cleaning, and drying

Refer to subclauses 7.2 to 7.4.

A.4 Measurement of specimen length

The length (L), in centimetres, of the specimen shall be measured with a relative combined standard uncertainty not to exceed 0,1 %.

A.5 Measurement of specimen diameter

The diameter (if it is a round wire) or two sides (if it is a rectangular wire) of the specimen shall be measured for the cross-sectional area measurement at five points along its length with combined standard uncertainty not to exceed 0,5 μm. Then the average cross-sectional area (A), in square centimetres, shall be calculated from those values obtained at the five points.

A.6 Measurement of specimen mass

The mass (M_W), in grams, of the specimen shall be measured on a balance with a manufacturer's specified uncertainty of ±0,1 mg or better.

A.7 Dissolving copper and measurement of dissolved specimen mass

The copper shall be measured in the same manner as in 7.6 and the cleaning and drying of the dissolved specimen shall be performed in the same manner as in 7.7.

The mass ($M_{Nb\text{-}Ti}$), in grams, of the filaments shall be determined in the same manner as Clause 7.8 of the main method.

A.8 Procedural repetition for the second specimen

The procedural steps in Clauses A.1 through A.6 shall be repeated on the second specimen. As soon as the method is qualified by a successful repetition, the repeated measurements on the

second specimen can be omitted in subsequent measurements. However, periodic re-qualification shall be performed every six months or after changes of equipment or personnel.

A.9 Calculation

Assuming the specific mass of the copper (ρ_{Cu}) 8,93 g/cm^3, the copper to superconductor volume ratio of Cu/Nb-Ti composite superconducting wires with copper mass method ($R_{Cu,m}$) shall be obtained using the following equation.

$$R_{Cu,m} = \frac{(M_W - M_{Nb-Ti})/\rho_{Cu}}{A \times L - (M_W - M_{Nb-Ti})/\rho_{Cu}} \quad (A.1)$$

NOTE 1 There may be large errors for the measurement of thin round wire and thin rectangular wire. So, care is taken for the measurement of those wires.

NOTE 2 For rectangular wire, the cross-sectional area (A), in square centimetres, is corrected according to the radius at the corners of the cross-sectional area, which is given in the specifications supplied by the manufacturers. In the case of rectangular wire, the uncertainty of the method in Annex A becomes worse if correction according to the radius at the corners is not taken into account.