INTERNATIONAL STANDARD

IEC 61788-5

First edition
2000-12

Superconductivity –

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

Supraconductivité –

Partie 5:
Mesure du rapport volumique matrice/supraconducteur – Rapport volumique cuivre/supraconducteur des composites supraconducteurs de Cu/Nb-Ti

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International Electrotechnical Commission 3, rue de Varembé Geneva, Switzerland
Telefax: +41 22 919 0300 e-mail: inmail@iec.ch IEC web site http://www.iec.ch

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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SUPERCONDUCTIVITY –

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

FOREWORD

1) The IEC (International Electrotechnical Commission) is a world-wide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of the IEC is to promote international cooperation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, the IEC publishes International Standard. Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International governmental and non-governmental organizations liaising with the IEC also participate in this preparation. The IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.

2) The formal decisions or agreements of the IEC on technical matters, prepared by technical committees on which all the National Committees have a special interest therein are represented, express, as nearly as possible, an international consensus of opinion on the subjects dealt with.

3) They have the form of recommendations for international use published in the form of standards, technical specifications, technical reports or guides and they are accepted by the National Committees in that sense.

4) In order to promote international unification, IEC National Committees undertake to apply IEC International Standards transparently to the maximum extent possible in their national and regional standards. Any divergence between the IEC Standards and the corresponding national or regional standard shall be clearly indicated in the latter.

5) The IEC provided no marking procedure to indicate its approval and cannot be rendered responsible for any equipment declared to be in conformity with one of its standards.

6) Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. The IEC shall not be held responsible for identifying any or all such patent rights.

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

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

FDIS	Report on voting
90/81/FDIS	90/87/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 3.

Annex A forms an integral part of this standard.

Annexes B, C and D are for information only.

The committee has decided that the contents of this publication will remain unchanged until 2005. At this date, the publication will be

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

A bilingual version of this standard may be issued at a later date.

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. If the specific mass of Nb-Ti and/or the fraction of Nb barrier are 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 superconductors

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 wire.

This test method is 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 conductors 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 in precision. 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 precision.

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 normative documents contain provisions which, through reference in this text, constitute provisions of this part of IEC 61788. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of IEC 61788 are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of IEC and ISO maintain registers of currently valid International Standards.

IEC 60050-815:2000, International Electrotechnical Vocabulary (IEV) – Part 815: Superconductivity

3 Terminology

For the purposes of this part of IEC 61788, the definitions given in IEC 60050-815 and the following definition are applicable.

3.1
copper to superconductor volume ratio
the ratio of the area of the copper stabilizing material to the area 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 in the nitric acid solution to dissolve only the copper.

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

Convert the ratio of masses to the ratio of volumes by dividing the respective masses of the copper and filaments by the respective specific mass of the copper and filaments.

5 Chemicals

The following chemicals shall be prepared for sample preparation.

– Nitric acid solution consisting of nitric acid (a mass fraction of 50 % to 65 % recommended) and distilled water
– Organic solvent
– Degreasing solvent
– Ethyl alcohol
– Distilled (pure) water

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

6 Apparatus

Draft chamber

Balance

A balance should have a measuring resolution of 0,1 mg.

Dryer or drying oven

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

– 300-ml beaker
– Watch-glass
– Plastic tweezers
– Filter papers
– Thermometer

Rubber gloves, protection spectacles and draft chamber

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.

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 in 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. 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.

7.5 Measurement of specimen mass

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, to an accuracy of 0,1 mg.

7.6 Repetition of mass measurement

After completion of this mass measurement (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 (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, 7.5 and 7.6 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 in equipment or personnel.

7.7 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 in 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, a 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.

NOTE 1 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.

NOTE 2 Use rubber gloves, protection spectacles and a pair of plastic tweezers during the treatment of the nitric acid solution.

NOTE 3 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.

NOTE 4 When mixing the solution, always add the nitric acid to the water.

7.8 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 strain 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.

NOTE 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) shall be 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.

7.9 Measurement of dissolved specimen mass and its repetition

When the specimen is cooled down to 35 °C or lower, using the balance, the specimen shall be weighed with an accuracy of 0,1 mg as in 7.5 and 7.6. A sheet of weighing paper shall be used for the measurement to avoid losing broken filaments (first measurement). After completion of the mass measurement described in 7.9, 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 (second measurement).

The difference in mass shall be within ±0,5 % between this 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 7.8, and then procedural steps shall be repeated again according to 7.9. 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.

7.10 Procedural repetition for second specimen

The procedural steps in 7.1 through 7.9 shall be repeated on the second specimen.

As soon as the method is qualified by a successful repetition, the repeated measurements on a 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.

$$\text{Copper to superconductor volume ratio} = \frac{(M_{\text{w}} - M_{\text{Nb-Ti}}) \times \rho_{\text{Nb-Ti}}}{M_{\text{Nb-Ti}} \times \rho_{\text{Cu}}} \quad (1)$$

where

M_{W} is the mass of the specimen (in grams);

$M_{\text{Nb-Ti}}$ is the mass of the Nb-Ti filaments (in grams);

ρ_{Cu} is 8,93, which is the specific mass of copper (in grams per cubic centimetre);

$\rho_{\text{Nb-Ti}}$ is the specific mass of the Nb-Ti filament (in grams per cubic centimetre).

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 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.

9 Precision and accuracy 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 the masses can be measured with an accuracy of 0,1 mg, the accuracy of the masses can be determined within ±0,1 % even for the specimen with a mass of 1 g and a copper to superconductor volume ratio of 10.

Precision is also affected by the specific mass of Nb-Ti. The value of the specific mass of Nb-Ti shall be determined within ±1 % by interpolation of the values listed in annex B (if not given by the wire manufacturer).

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 the precision.

If the specific mass of Nb-Ti and/or fraction of Nb barrier are unknown (use of annex A), the measurement accuracy of the specimen size shall be better than 0,2 %.

In the case of rectangular wire, it shall be noted that the accuracy of the method in annex A becomes worse if correction according to the radius at the corners is not taken into account.

The overall precision of the main method shall be 2 % based on a C.O.V. of 0,8 % according to round robin tests made to establish this standard.

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

If the specific mass of Nb-Ti and/or the fraction of the Nb barrier are unknown, the copper to superconductor volume ratio shall be measured in the following manner.

A.1 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.2 Measurement of specimen length

The length (L), in centimetres, of the specimen shall be measured with an accuracy of ±0,2 % or better.

A.3 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 an accuracy of ±1 µm. Then the average cross-sectional area (A), in square centimetres, shall be calculated from those values obtained at the five points.

A.4 Measurement of specimen mass

The mass (M_W), in grams, of the specimen shall be measured with an accuracy of 0,1 mg.

A.5 Dissolving copper and measurement of dissolved specimen mass

The copper shall be removed in the same manner as the one in the main method.

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

A.6 Calculation

Assuming the specific mass of the copper (ρ_{Cu}) 8,93 g/cm^3, the copper to superconductor volume ratio shall be obtained using the following equation.

$$\text{Copper to superconductor volume ratio} = \frac{(M_W - M_{Nb\text{-}Ti})/\rho_{Cu}}{A \times L - (M_W - M_{Nb\text{-}Ti})/\rho_{Cu}} \qquad \text{(A.1)}$$

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

NOTE 2 For rectangular wire, the cross-sectional area (A), in square centimetres, needs to be corrected according to the radius at the corners of the cross-sectional area, which is given in the specifications supplied by the manufacturers.

Annex B
(informative)

Specific mass of Nb-Ti

Nb-Ti fraction	Specific mass g/cm^3
Nb	8,57
Nb - 43,2 wt % Ti	6,16
Nb - 45,0 wt % Ti	6,09
Nb - 46,5 wt % Ti	6,04
Nb - 47,0 wt % Ti	6,02
Nb - 48,0 wt % Ti	5,98
Nb - 53,5 wt % Ti	5,76
Nb - 55,0 wt % Ti	5,70
Ti	4,51

NOTE 1 The specific mass of the Nb-Ti alloy depends not only on its composition but also on other parameters: amount of cold work, impurities, phase condition, and so on.

NOTE 2 Precision = ±1 %. Additional digits are provided for more precise interpolation.