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Superconductivity Teh STANDARD PREVIEW Part 12: Matrix to superconductor volume ratio measurement – Copper to non-

copper volume ratio of Nb₃Sn composite superconducting wires

IEC 61788-12:2013

Supraconductivité //standards.iteh.ai/catalog/standards/sist/494e1da4-741b-4d96-91cc-Partie 12: Mesure du rapport volumique matrice/supraconducteur – Rapport volumique cuivre/non-cuivre des fils en composite supraconducteur Nb₃Sn





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Part 12: Matrix to superconductor volume ratio measurement – Copper to noncopper volume ratio of Nb₃Sn composite superconducting wires

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

SUPERCONDUCTIVITY -

Part 12: Matrix to superconductor volume ratio measurement – Copper to non-copper volume ratio of Nb₃Sncomposite superconducting wires

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International Standard IEC 61788-12 has been prepared by IEC technical committee 90: Superconductivity.

This second edition cancels and replaces the first edition published in 2002. It constitutes a technical revision. The main revision is the addition of two new annexes, "Uncertainty considerations" (Annex H) and "Uncertainty evaluation in the test method of the copper to non-copper volume ratio of Nb₃Sn composite superconducting wires" (Annex I).

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

| FDIS | Report on voting |
|-------------|------------------|
| 90/322/FDIS | 90/325/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

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INTRODUCTION

The copper to non-copper volume ratio of superconducting wires serves as an important numeric value used when determining the critical current density and its stability, which are two of the important characteristics of superconducting wires. This standard is concerned with the standardization of the test method for the copper to non-copper volume ratio of copper stabilized Nb₃Sn multi-filamentary composite superconducting wires (hereinafter referred to as Cu/Nb₃Sn wires).

Cu/Nb₃Sn wires can be classified into four types according to the layout of the stabilizer as shown in Annex G: the external stabilizer type, the internal stabilizer type, the distributed stabilizer type and the contiguous stabilizer with distributed barrier type. The test method specified by this standard may be applicable to a type whose cross-section is of the external stabilizer or the internal stabilizer type regardless of the production process employed.

With regard to the internal stabilizer type, the internal structure of some Cu/Nb_3Sn wires prevents copper from being dissolved and removed. This precludes the application of the copper mass method, unlike with copper matrix Nb-Ti superconducting wires. New methods are therefore needed, as detailed in the following:

- the paper mass method, where a photo of the cross-section of the wire being measured is traced onto tracing paper, or a copy is made of the photo using a copying machine; the paper is then cut out into different portions to measure the mass of each piece of paper;
- the image processing method, where the image of the photo of the cross-section is digitized and the areas are analyzed with software;
- the copper mass method, where the copper of the specimen is dissolved in nitric acid solution to leave only the non-copper portion, and to measure the mass of the specimen and the non-copper portion of specimen. IEC 61788-12:2013

This standard is concerned with the paper mass method which is adopted more generally. As supplementary methods, the image processing method and the copper mass method adopted for Cu/Nb₃Sn wires are specified in Annex A and Annex B, respectively. The method using a planimeter is specified in Annex C. In Annex D an example of a polishing method is also specified.

SUPERCONDUCTIVITY -

Part 12: Matrix to superconductor volume ratio measurement – Copper to non-copper volume ratio of Nb₃Sn composite superconducting wires

1 Scope

This part of IEC 61788 describes a test method for determining the copper to non-copper volume ratio of Cu/Nb₃Sn wires.

The test method given hereunder is applicable to Nb₃Sn composite superconducting wires with a cross-sectional area of 0,1 mm² to 3,0 mm² and a copper to non-copper volume ratio of 0,1 or more. It does not make any reference to the filament diameter; however, it is not applicable to those superconducting wires with their filament, Sn, Cu-Sn alloy, barrier material and other non-copper portions dispersed in the copper matrix or those with the stabilizer dispersed. Furthermore, the copper to non-copper volume ratio can be determined on specimens before or after the Nb₃Sn formation heat treatment process.

The Cu/Nb₃Sn wire has a monolithic structure with a round or rectangular cross-section.

Though uncertainty increases, this method may be applicable to the measurement of the copper to non-copper volume ratio of the Cu/Nb₃Sn wires whose cross-section and copper to non-copper volume ratio fall outside the specified ranges.

This test method may be applied to the 78 composite superconducting wires after some appropriate modifications and ards.iteh.ai/catalog/standards/sist/494e1da4-741b-4d96-91ccfb5e39cb803d/iec-61788-12-2013

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 (all parts), International Electrotechnical Vocabulary (available at http://www.electropedia.org

IEC 61788-5, Superconductivity – Part 5: Matrix to superconductor volume ratio measurement – Copper to superconductor volume ratio of Cu/Nb-Ti composite superconductors

3 Terms and definitions

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

3.1

copper to non-copper volume ratio

ratio of the volume of the copper stabilizing material to the volume without copper consisting of Cu/Nb₃Sn wires

4 Principle

The principle of this method is described in the following. A photo of the polished cross-section of the sample wire shall be taken with a metallograph. This photo is traced onto tracing paper, or a copy is made of the photo using a copy machine. The tracing paper or copy is then cut out into different portions to measure the mass of each piece of paper. The copper to non-copper volume ratio can be obtained from the ratio of the paper mass of both portions.

5 Apparatus

The apparatus required for the test includes the following:

- metallograph;
- photomicrographic camera;
- polishing machine;
- balance;

6.1

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

• scissors or cutter.

6 Measurement procedure

Preparation of specimen TANDARD PREVIEW

6.1.1 General (standards.iteh.ai)

The specimen shall be prepared from $a_{\rm C}u/Nb_{3}$ Sn_wire before or after the Nb₃Sn generation heat treatment process. Two specimens shall be cut out of a Cu/Nb₃Sn wire being measured.

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NOTE In the case of measuring an internal tin processed wire before the treatment, the stabilizer copper is carefully distinguished from copper in other parts.

6.1.2 Procedures

6.1.2.1 Mold

The two specimens shall be molded at the same time for polishing. As the molding material, an appropriate resin shall be used to embed the specimen for observation through the metallograph. When molding, it shall be carefully checked that the cross-section of the specimen is at right angles to the polishing surface as much as possible.

6.1.2.2 Polishing

The specimen shall be polished using emery paper and buffed using an abrasive material. A microscope shall be used to check that the polished surface is smooth enough to ensure good photographing, especially the boundary between the copper and non-copper portions and the periphery of the sample. The surface shall be re-polished, if these areas are not clear because of abrasive scratches. An example of the polishing method is specified in Annex D.

6.1.2.3 Cleaning and drying

The polished specimen shall be cleaned using running water, distilled water, acetone or ethyl alcohol. It shall be checked that the cleaning agent does not dissolve the resin in which the specimen is embedded. An ultrasonic cleaning machine may be used if necessary. After cleaning, the specimen shall be dried with chilled or hot air to prevent the polished surface from oxidizing or discoloring.

6.2 Measurement

6.2.1 Photo of cross-section

A black-and-white or color picture of the cross-section shall be taken using the metallograph.

The magnification shall be selected for the entire cross-section of the specimen to fit within the size of the photo. A photomicrographic camera with as much depth of focus as possible shall be used, so that the boundary between copper and non-copper portions and the periphery of the specimen appear clear and vivid on the photo.

6.2.2 Transfer

The image of the cross-section shall be traced on tracing paper so that the copper portion and non-copper portion can be separated.

As an alternative method, a copy of the photo of the cross-section shall be made using a copying machine. A zoom ratio of the copier that will fit the image in a sheet of paper and allow the cutting work to be done easily shall be selected. If a copy is to be made using a copy machine, a copy of a scale shall be made at the same time, to check that the copier distortion is within ± 1 % (see Annex F).

6.2.3 Cutting

Scissors or a cutter shall be used to cut the tracing paper or the copy into the copper and non-copper portions.

(standards.iteh.ai) Measurement of paper mass

6.2.4

The first specimen shall be measured twice 61788-12:2013

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The paper mass of the copper portion and non-copper portion shall be measured with a combined standard uncertainty not to exceed 0,1 mg. Each portion shall be measured twice and the average of the two measurements shall be reported.

During the measurement, caution shall be taken to ensure that the measurement is not affected by humidity. If the mass continues to change, the specimen shall remain in the measurement chamber for about 10 min before resuming the measurement sequence.

6.3 Test procedure for the second specimen

The steps given under 6.2 shall be repeated to measure the paper mass for the second specimen.

6.4 Paper mass

The paper mass of the copper portion (M_{Cu}) to that of the non-copper portion (M_{non}) shall be obtained by averaging the paper mass measured at steps 6.2 and 6.3.

7 Calculation of results

For each measurement taken in 6.2 and 6.3, the copper to non-copper volume ratio shall be obtained from the ratio of the paper mass of the copper portion to that of the non-copper portion. Copper to non-copper volume ratio is expressed as M_{Cu}/M_{non}

The ratio shall be rounded to two decimal places.

Uncertainty of the test method 8

The uncertainty of this test method is affected by the sag of the specimen occurring from polishing, transfer to tracing paper, distortion of the copying machine and uncertainty in cutting out portions from the paper. The relative combined standard uncertainty of this method shall not exceed 2,5 % (using a coverage factor of k = 1) as shown in Clause I.1.

9 **Test report**

9.1 Copper to non-copper volume ratio

The report shall contain the following information:

- a) copper to non-copper volume ratio of each specimen;
- b) wire diameter or size of the cross-section if it is a rectangular shape;
- c) whether the specimens had or had not received the Nb₃Sn generation heat treatment.

The report shall contain the following information if known:

- d) manufacturing method;
- e) configuration of the copper matrix;
- f) photo of cross-section;
- g) measurement conditions and information ARD PREVIEW
- h) raw measured data;
- information of measurement equipment. i)

Identification of test specimen IEC 61788-12:2013 9.2

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

- a) name of the manufacturer of the specimen;
- b) identification number;
- c) billet number.

Annex A

(normative)

Measurement – Image processing method

A.1 Method

The following details describe the method that can be used to digitize the image on the cross-sectional photo (image processing method).

- a) Following the steps from 6.1 through 6.2.1, photos of the cross-section of the specimens shall be taken.
- b) Using a scanner, the image of the cross-section photos shall be captured in a personal computer.
- c) Using image analysis software, the number of pixels on the copper portion and non-copper portion shall be determined.
- d) The copper to non-copper volume ratio of the specimen shall be determined using the following equation:

$R_{Cu,i} = \frac{N_{Cu}}{N_{non}}$ **iTeh STANDARD PREVIEW**(A.1)

where

 R_{cui} is the copper to non-copper volume ratio with image processing method;

- $N_{\rm Cu}$ is the number of pixels on the copper portion;
- N_{non} is the number of pixels on the non-copper portion4e1da4-741b-4d96-91cc-

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A.2 Test report

The following information shall be reported in addition to the data listed in Clause 9: image analysis software used.

NOTE 1 Measurement uncertainty of the image processing method is determined by the quality of image of the photo of cross-section. What is necessary to ensure a given level of uncertainty is taking a clear image of the specimen cross-section with a well-polished condition.

NOTE 2 Reproducibility of the measurements taken through the image processing method applied to the image captured from the same position at the same magnification is estimated by a relative combined standard uncertainty not to exceed 5 %.

Annex B

(normative)

Measurement – Copper mass method

B.1 Method

The following describes the application of the copper mass method (see IEC 61788-5), which is employed for measuring the copper to superconductor volume ratio of Nb-Ti superconducting wires, to Nb₃Sn. This method can be applied only to the external stabilizer type Cu/Nb₃Sn wire with a round cross-section that exhibits a nature whereby copper dissolves in nitric acid. Nevertheless, it shall be avoided to apply this method to such wires if they have barriers that can be broken in the process of dissolving by nitric acid.

a) A specimen with a length of 300 mm to 500 mm shall be cut and the mass (M_1) , length (L) and diameter (D) of the specimen shall be determined. The diameter shall be measured at five equally divided points and, taking the average of the measurements, the volume (V_1) shall be calculated:

$$V_1 = \pi (D/2)^2 L$$
 (B.1)

- b) The copper of the specimen shall be dissolved in nitric acid solution completely to leave only the non-copper portion. At this time, the specimen shall be rinsed quickly with water once the copper has dissolved, thereby minimizing the amount of bronze dissolved.
- c) The specimen shall be dried completely after rinsing.
- d) The mass (M_2) of non-copper portion shall be measured.
- e) The volume (V₂) of the copper portion shall be calculated using 8,93 g/cm³ as the specific mass of copper https://standards.iteh.ai/catalog/standards/sist/494e1da4-741b-4d96-91cc-

$$\frac{\text{fb}5\text{e39cb}803d/\text{iec-}61788-12-2013}{V_2 = (M_1 - M_2) / 8,93}$$
(B.2)

f) The copper to non-copper volume ratio with copper mass method ($R_{Cu,c}$) shall be calculated from the volume (V_1) of the specimen and volume (V_2) of the copper portion.

$$R_{\rm Cu,c} = V_2 / (V_1 - V_2) \tag{B.3}$$

g) The relative combined standard uncertainty of this method shall not exceed 2,5% (using a coverage factor of k = 1) as shown in Clause I.3.

B.2 Test report

The following information shall be reported in addition to the data listed in Clause 9: the necessary information according to the test report in IEC 61788-5.

Annex C

(normative)

Measurement method using planimeter

C.1 Method

The following details describe the method using an analogue or a digital planimeter.

- a) According to steps 6.1 to 6.2.1, a photo of the cross-section shall be taken.
- b) A copy of the photo of the cross-section shall be made using a copying machine. A zoom ratio of the copier shall be selected so that the size of the enlarged image is more than 120 mm in width and within a sheet of paper.
- c) The values of cross-sections for copper and non-copper parts shall be obtained using a planimeter. Measurement with 5 turns of the planimeter to integrate the area is recommended in order to reduce the uncertainty. The measurement shall be carried out twice for the same photo, and the average value shall be the cross-sectional area if the measured values are within 5 %. If this value is more than 5 %, the measurement shall be carried out again.

NOTE In the case of relative combined standard uncertainty of a planimeter within 0,5 %, either an analog or a digital planimeter apparatus can be used.

d) In the case of an external stabilizer type, the cross-sectional area of the copper part shall be obtained by subtracting that of the non-copper part from the whole area of the specimen. In the case of an internal stabilizer type, the cross-sectional area of the non-copper part shall be obtained by subtracting that of the copper part from the whole area.

C.2 Test report

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The following information shall be reported in addition to the data listed in Clause 9: type of planimeter and zoom ratio of the copy used.