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Welding — Micro joining of 2nd generation high temperature superconductors —

Part 1: General requirements for the procedure

Soudage — Micro-assemblage des supraconducteurs à haute température de 2ème génération —

Partie 1: Exigences générales pour la procédure

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Foreword

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This document was prepared by Technical Committee ISO/TC 44, *Welding and allied processes*, Subcommittee SC 10, *Quality management in the field of welding*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

A list of all parts in the ISO 17279 series can be found on the ISO website.

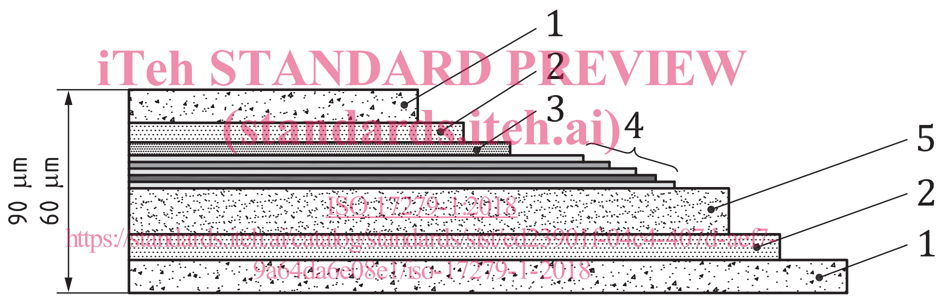
Introduction

The increasing use of 2nd generation high temperature superconductors (2G HTSs) and invention of resistance-free joining on 2G HTSs have created the need for this document in order to ensure that joining is carried out in the most effective way and that appropriate control is exercized over all aspects of the operation. ISO standards for micro-joining and joint evaluation procedure are accordingly essential to get the best and uniform quality of 2G HTS joint.

The technique in this document regarding resistance-free micro-joining is patent-registered and was reported to patent.statements@iso.org using the “Patent Statement and Licensing Declaration Form”.

A superconductor is a material that conducts electricity without resistance and has diamagnetism below critical temperature, T, critical magnetic field, B_c, and critical current density, J_c. Once set in motion, electrical current flows forever in a closed loop of superconducting material under diamagnetism.

A 2G HTS consists of multi-layers and its total thickness is around between 60 µm and 100 µm with or without surrounding copper stabilizer. The superconducting layer made from $ReBa_2Cu_3O_7$-x (ReBCO, abbreviated term of $ReBa_2Cu_3O_{7-x}$) is only between 1 µm and 2 µm thick depending on manufacturer’s specifications. Re stands for Rare Earth materials, of which gadolinium, yttrium and samarium are used for 2nd generation high temperature superconducting materials. Figure 1 shows schematic drawing of typical multiple layers with surrounded copper stabilizer, and the constituents and thicknesses of each layer in the 2G HTS. The two layers of No. 1 in Figure 1 does not exist in stabilizer-free 2G HTS.



Key

1 20 µm Cu stabilizer

2 2 µm Ag overlayer

3 between 1 µm and 2 µm ReBCO super-conducting layer

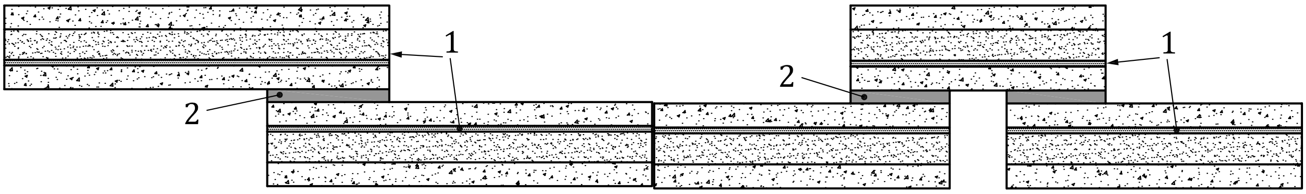
4 5 buffing layers (total 160 nm)

5 50 µm hastelloy substrate

NOTE Not to scale.

Figure 1 — Typical 2G HTS multi-layers, and the constituents and thicknesses of each layer

Currently soldering, brazing or any filler is applied in superconducting industry as shown in Figure 2, which shows high electrical resistance at the joint providing fatal flaw in the superconductor.



a) Lap joint **b) Bridge joint**

Key

1 superconducting layer

2 solder

Figure 2 — Soldering to join 2G HTS

However, this document focuses on the direct autogenous joining of between 1 μm and 2 μm-thick superconducting layers of 2G HTSs as shown in Figure 3 without filler metals and recovery of superconducting properties by oxygenation annealing process, which shows almost no electrical resistance at the joint.

a) Lap joint **b) Bridge joint**

Key

1 superconducting layer

Figure 3 — Direct autogenous joining of two superconducting layers of 2G HTSs for superconducting joint

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Welding — Micro joining of 2nd generation high temperature superconductors —

Part 1: General requirements for the procedure

1 Scope

This document provides concepts, specification and qualification of 2G HTS joining procedure. A welding procedure specification (WPS) is needed to provide a basis for planning joining operations and for quality control during joining. Joining is considered as a special process in the terminology of standards for quality systems. Standards for quality systems usually require that special processes be carried out in accordance with written procedure specifications. This has resulted in the establishment of a set of rules for qualification of the joining procedure prior to the release of the WPS to actual production. This document defines these rules.

This document does not cover soldering, brazing or any fillers, which are currently available in the industry. It can be applied for joining of all kinds of 2G HTSs.

This document does not apply to 1st Generation Bismuth Strontium Calcium Copper Oxide (1G BSCCO) type HTS and Low Temperature Superconductor (LTS) Joining.

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.

ISO 15607:2003, *Specification and qualification of welding procedures for metallic materials — General rules*

ISO 17279-2, *Welding — Micro-joining of 2nd generation high temperature superconductors — Part 2: Personnel qualification for micro-joining and testing*

ISO/TR 25901 (all parts), *Welding and related processes — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TR 25901 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
high temperature superconductor
HTS
superconducting material with critical temperature higher than liquid nitrogen boiling point

3.2
low temperature superconductor
LTS
superconducting material with critical temperature lower than liquid nitrogen boiling point

3.3
2nd generation high temperature superconductor
2G HTS
superconducting material with critical temperature higher than liquid nitrogen boiling point, made of rare earth and other elements like barium, copper and their oxides

Note 1 to entry: A first generation high temperature superconductor (1G HTS) is a superconducting material with critical temperature higher than liquid nitrogen boiling point, made of bismuth strontium calcium copper oxides.

3.4
pressurized partial micro-melting diffusion
partial micro-melting of the two faying surfaces of 1 µm- to 3 µm-thick superconducting layers and atoms diffusion in partially micro-molten pool and solid state of the superconducting layers by pressurized force

3.5
oxygenation annealing
process to restore the oxygen stoichiometry in an oxygen-rich environment and to recover superconducting properties

Note 1 to entry: Structure and superconducting properties of 2G HTS are strongly affected by the oxygen stoichiometry. Joining of 2G HTS at high temperature induces oxygen out-diffusion causing a phase change from a superconducting orthorhombic phase to a non-superconducting tetragonal phase.

3.6
pressurized solid-state diffusion
atoms diffusion in solid state of the two faying surfaces of 1 µm- to 3 µm-thick superconducting layers with pressurized force

3.7
bridge joint
joint with a third 2G HTS piece overlapped as a bridge on top of two 2G HTS pieces

Note 1 to entry: See Figure 3 and Figure 5.

4 Symbols and abbreviated terms

The abbreviatied terms listed in ISO 15607:2003, Table 1, relevant to joining procedure for 2G HTS, shall apply.

5 Requirements

5.1 Joint design

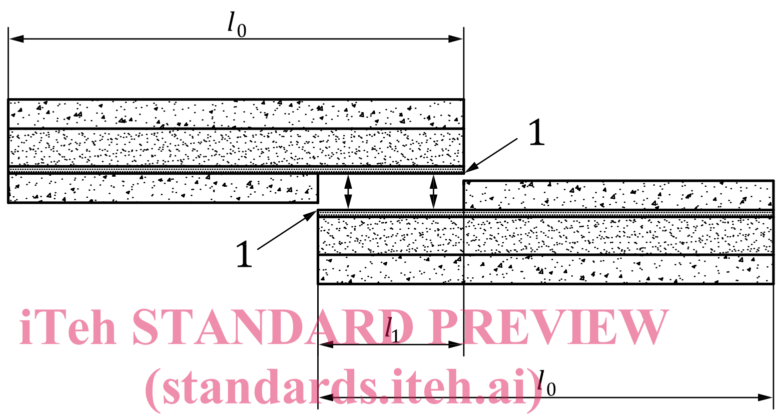
5.1.1 General

The joint shall be designed in accordance with defined requirements that support the end use of the product. Documentation shall clearly define the essential information of the joint and any special requirements, e.g. fracture critical, durability critical, mission critical, or safety critical, that are imposed over and above the general requirements. Essential process controls shall be defined to substantiate that all design requirements can be met by the joints that were produced in accordance with welding procedure specification (WPS) and testing and inspection requirements.

The joint design shall take into account the necessary material property data. There are basically two types of joint alignments (lap and bridge) and the suitable alignment can be selected depending on user's convenience. The two types of joint alignments for joining of 2G HTS are shown in Figure 3.

5.1.2 Lap joint

Figure 4 shows schematic sketch of lap joint design for welding procedure qualification as described in 5.3. All lengths, l_0, and l_1, should be specified in the procedure qualification report (PQR). As joint length, l_1, is increased, effective contact surface areas between two superconducting layers are increased, probabilities of atoms inter-diffusion between the two layers for the joining are increased, and accordingly joint strengths (tensile and bending) are increased. The Cu stabilizers, if included, and Ag overlayers on top of the superconducting layers shall be perfectly removed for direct contact of two superconducting layers and micro-joining. This process is for not contaminating superconducting layers during micro-joining.



Key

1 superconducting layer

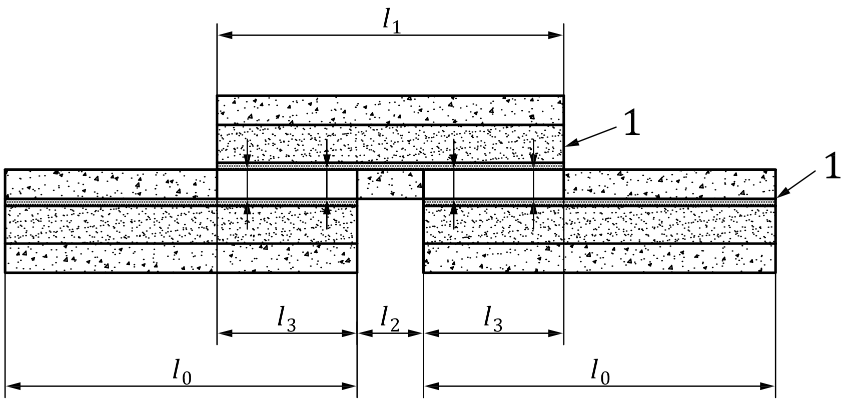
l_0 60 mm; length of two parts

l_1 40 mm; overlap (joint length)

Figure 4 — Lap joint

5.1.3 Bridge joint

Figure 5 illustrates the joint design for welding procedure qualification as described in 5.3. The distance, l_2, between two 2G HTS parts in Figure 5 may be ranged from "0" to over 10 cm depending on joint design. The lengths of l_0 in Figure 5 should be the same. All lengths, l_0, l_1, l_2, and l_3, should be specified in the procedure qualification report (PQR). As joint lengths, l_3, are increased, effective contact surface areas between two superconducting layers are increased, probabilities of atoms inter-diffusion between the two layers for the joining are increased, and accordingly joint strengths (tensile and bending) and superconductivity are increased. The Cu stabilizers, if included, and Ag overlayers on top of the superconducting layers shall be perfectly removed for direct contact of two superconducting layers and joining. This process is to avoid contaminating the superconducting layers during joining.



Key

1 superconducting layer

l_0 50 mm; length of two parts at bottom

l_1 40 mm to 50 mm; length of bridge part

l_2 0 mm to 10 mm; distance between parts to join

l_3 20 mm; overlap (joint length)

Figure 5 — Bridge joint

5.2 Equipment

The equipment shall be adequate for the application concerned. Joining equipment shall be capable of producing joints that meet the acceptance criteria specified in 5.9. Joining equipment shall be maintained in good condition and shall be repaired or adjusted when a joining operator, inspector or joining coordinator is concerned about the capability of the equipment to operate satisfactorily.

After installation of new or refurbished equipment or developing the new equipment, appropriate tests shall be performed. Such tests shall verify the equipment functions correctly.

Reproducibility tests shall be performed to demonstrate that the joining equipment can repeatedly produce joints that meet the acceptance levels in 5.9. The reproducibility test shall be performed in accordance with a WPS that is used in production for that machine. A minimum of three test joins shall be made and found satisfactory. Reproducibility tests shall be carried out when any of the following occurs:

— critical component(s) of the equipment is (are) damaged, repaired, or replaced;

— equipment is dislodged or moved in a manner for which it was not designed;

— stationary equipment is moved from one location to another.

The manufacturer shall have a documented plan for equipment maintenance. The plan shall ensure that maintenance checks are performed on the equipment that controls variables listed in the relevant WPS. The maintenance plan may be limited to those items that are essential for producing joints that meet the quality requirements of this document.

The joining tools, if any, that are used in production shall be permanently marked for identification prior to use. Before joining, the joining tools, if any, shall be clean and sufficiently free of contaminants (e.g. oil, grease or dirt) that can have a detrimental effect on joint quality. The correct tool geometry is critical for producing a quality joint. Because the joining tool wears with use, it shall be inspected for wear at appropriate intervals and in accordance with a written procedure. Before joining, parts and pieces that contact the joining part shall be clean and sufficiently free of contaminants (e.g. oil, grease, and dirt) that can have a detrimental effect on the joint.

Defective equipment shall not be used.

NOTE Joining machine and oxygenation annealing machine are rarely commercially available. It is the responsibility of each company or organization performing joining to develop the machines, if suitable commercial machines are not available.

5.3 Welding procedure qualification

The manufacturer shall be fully responsible for the specification and performance of micro-joining and oxygenation-annealing for recovery of superconductivity which is determined by the manufacturer. A record of the micro-joining and oxygenation-annealing shall be made before, during and after the process.

The following information shall be specified for each joint in WPS:

a) supplier's product specification of 2G HTS;

b) superconducting layer material specification;

c) pre-joint surface preparation, including complete removal of Cu stabilizer if included and Ag overlayer;

d) joint type (lap, bridge or other joint type if other than lap or bridge);

e) specimen dimensions including joint length (l_0, l_1, l_2, l_3 of Figure 4 or Figure 5);

f) final joint finishing and configuration (any reinforcement for increasing joint strength);

g) joint dimensions; the dimensions of the joint on the welding procedure specification (WPS) shall be the final dimensions.

A form of WPS is shown in Annex D.

5.4 Micro-joining and oxygenation-annealing process

5.4.1 General

The requirements for the specification of micro-joining and oxygenation-annealing process for 2G HTS are specified. Qualification of micro-joining and oxygenation-annealing procedures shall be performed prior to production joining. The manufacturer shall prepare a welding procedure qualification report (WPQR) and a welding procedure specification (WPS) and shall ensure that it is applicable for production using experience from previous production jobs and the general knowledge of joining technology.

The superconducting layer is made from ReBCO ($ReBa_2Cu_3O_{7-x}$) in which the molar ratio of Re:Ba:Cu is 1:2:3 and the mole fraction (7-x) of oxygen (O), is typically in the range of 6,4 to 7. In oxygen partial pressure of around 21,5 kPa in ambient air, a superconducting joint is rarely obtained without melting the Ag overlayer that protects the superconducting layer because the melting point of ReBCO is higher than that of Ag. However, a successful superconducting joint can be obtained without deformation of the ReBCO layer by reducing the oxygen partial pressure, which helps reduce the melting point of ReBCO. Thus, joining in vacuum condition is required. On the other hand, joining under a vacuum, which is controlled by the out-diffusion of oxygen, can degrade the superconducting properties. The structure and superconducting properties of ReBCO are strongly affected by the oxygen stoichiometry, hence oxygen deficiencies cause a phase change from a superconducting orthorhombic phase to a non-superconducting tetragonal phase. This phase transition is dependent on the temperature and oxygen partial pressure. Moreover, the phase transition in an atmosphere containing oxygen is reversible with changes in temperature. However, it is irreversible in the vacuum state resulting in a tetragonal phase that remains stable after heat-treatment at high temperatures. Therefore, it is important to develop oxygenation annealing at elevated temperatures around just under transition temperature

