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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

Reference number
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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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.

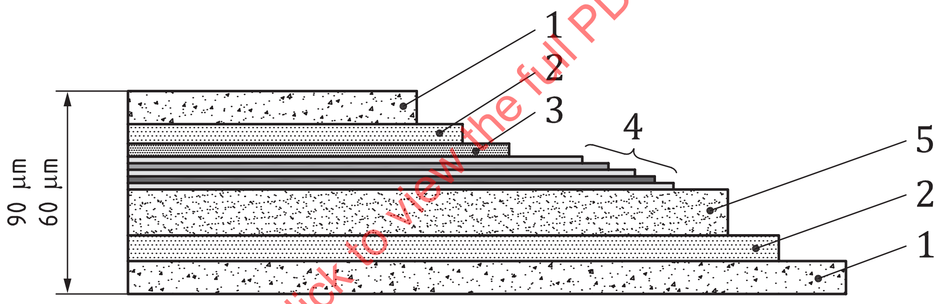
Introduction

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

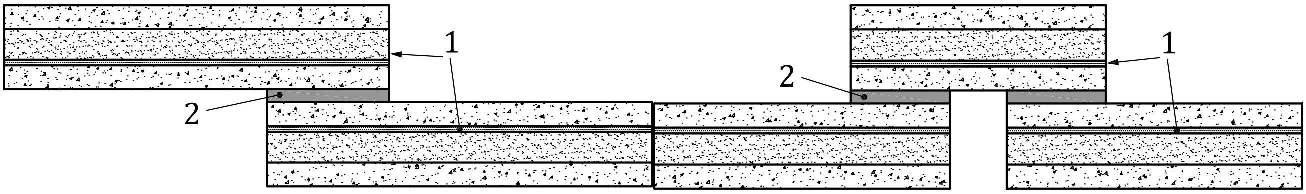
4 5 buffing layers (total 160 nm)

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**

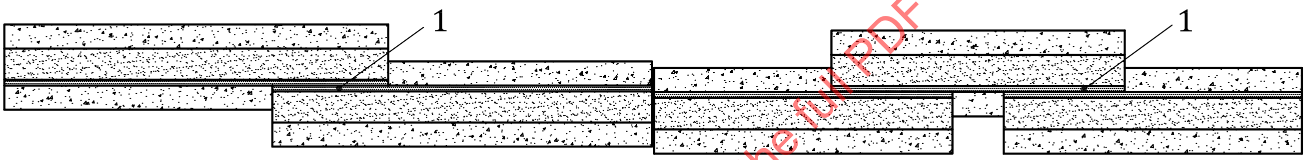
Key

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

The International Organization for Standardization (ISO) draws attention to the fact that it is claimed that compliance with this document may involve the use of patents concerning 2G HTS resistance-free joining. ISO takes no position concerning the evidence, validity and scope of this patent right. The holders of these patent rights have assured ISO that they are willing to negotiate licenses under reasonable and non-discriminatory terms and conditions with applicants throughout the world. In this respect, the statement of the holders of these patent rights is registered with ISO. Information may be obtained from:

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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

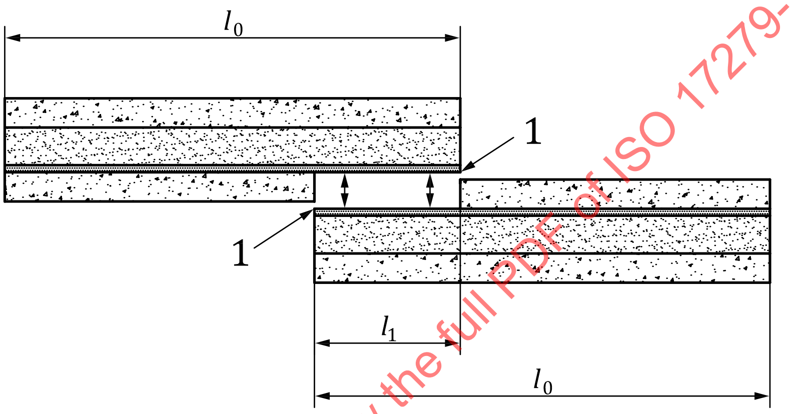
5.1.1 General

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.



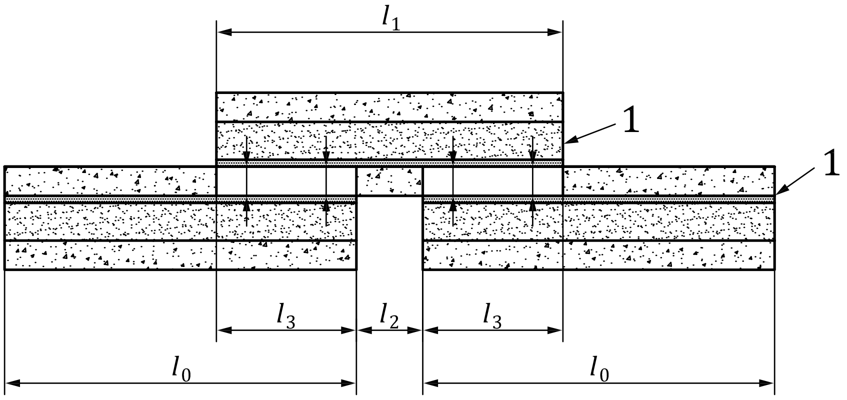
Key

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.

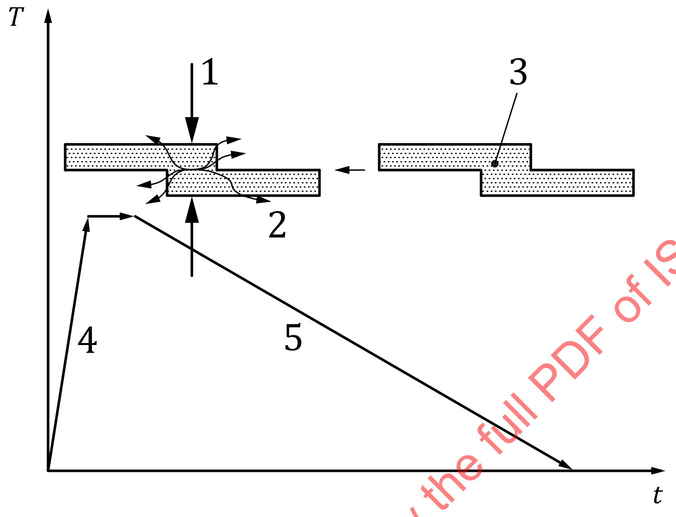
5.4 Micro-joining and oxygenation-annealing process

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 of superconducting orthorhombic phase to a non-superconducting tetragonal phase in an oxygen rich environment, to restore the oxygen stoichiometry and hence superconducting properties at the joint.

Figures 6 and 7 illustrate micro-joining process and oxygenation-annealing process cycles, respectively. Micro-joining takes place in direct contact of two superconducting layers with extremely short period of time. However, oxygenation-annealing takes place for hours or days depending on the furnace (chamber) capacity, oxygen environment, purity and concentration, joint types and quality. Micro-joining and oxygenation-annealing may be performed in separate equipment. Separate operation of oxygenation-annealing has advantages to anneal many specimens in one time depending on chamber size. Figure 8 shows micro-joining and oxygenation-annealing processes in the same chamber and with continuous operation.

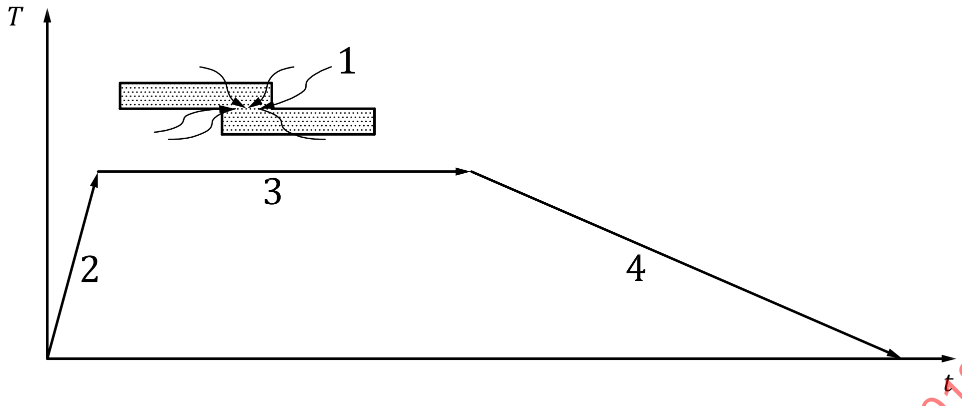


Key

1 application of heat and pressure on lapped pieces
2 oxygen out-diffusion from superconducting layer
3 peel-off stabilizers and/or overlayers on top of the superconducting layer and lapped
4 fast heating
5 fast-moderate cooling

NOTE Heat and pressure under vacuum during joining.

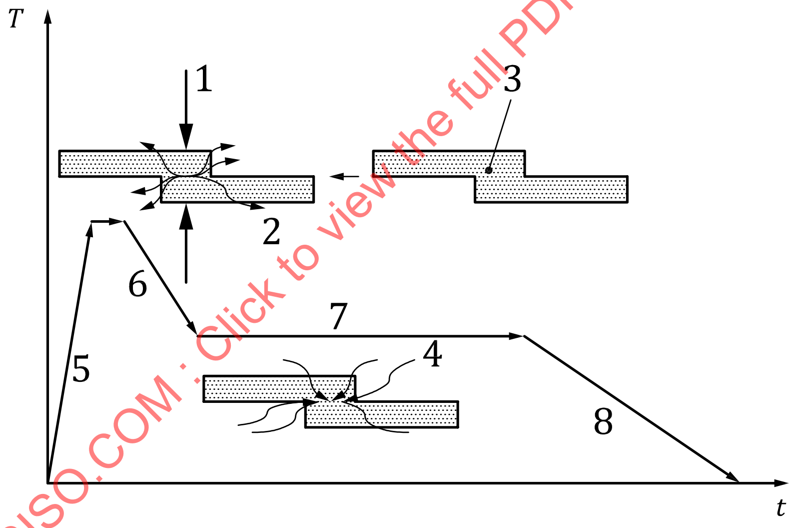
Figure 6 — Cycle of micro-joining process in joining chamber



Key

1	oxygen in-diffusion into the superconducting layer during oxygenation annealing	3	oxygenation annealing
2	moderate heating	4	slow cooling

Figure 7 — Cycle of oxygenation-annealing process under pressurized oxygen environment in oxygenation-annealing chamber



Key

1	application of heat and pressure on lapped pieces	5	fast heating
2	oxygen out-diffusion from superconducting layer	6	fast-moderate cooling
3	peel-off stabilizers and/or overlayers on top of the superconducting layer and lapped	7	oxygenation annealing
4	oxygen in-diffusion into the superconducting layer during oxygenation annealing	8	slow cooling

Figure 8 — Cycle of micro-joining and oxygenation-annealing processes in the same chamber and with continuous operation

A preliminary welding procedure specification (pWPS) shall be used as the basis for the establishment of a welding procedure qualification test (WPQT) and a welding procedure qualification record (WPQR). For testing the pWPS, see the test methods given in ISO 17279-3,[1]

[1] Under preparation.

Examples of a micro-joining and oxygenation-annealing procedure, pWPS form, WPQR and WPS are shown in Annex A, B, C and D, respectively.

5.4.2 Technical content of a pWPS and WPS

The following information, as a minimum, shall be included in a pWPS and WPS:

a) manufacturer's information: identification of the manufacturer, the pWPS and WPS;

b) 2G HTS superconducting material type (YBCO, GdBCO, SmBCO, etc);

c) 2G HTS parent material dimensions:

  1) total thickness of the 2G HTS members;

  2) thickness of the superconducting layer comprising the joint;

  3) width of 2G HTS members;

d) equipment identification: model, serial number, manufacturer;

e) joint design: sketch of the joint design and dimensions and configuration of the joint;

f) joint preparation and cleaning methods:

  1) Cu stabilizer and/or Ag overlayer removal;

  2) materials (chemical etchants, etc) to remove Cu stabilizer and/or Ag overlayer;

  3) any specific;

g) joining details:

  1) chamber vacuum level, heat treatment cycle sketch and heating rate to peak temperature;

  2) pressure applied to the specimen, peak temperature, and dwell time at peak temperature;

  3) cooling rate from peak temperature to room temperature;

  4) details of any changes and/or any specifics.

h) oxygenation annealing details:

  1) heat treatment cycle sketch, oxygen flow rate, chamber internal pressure;

  2) heating rate, peak temperature, and dwell time at oxygenation-annealing temperature;

  3) cooling rate from oxygenation-annealing temperature to room temperature;

  4) designation (if any), manufacturer, name and purity of oxygen gas;

  5) details of any changes and or any specifics;

i) joint reinforcement details: methods, materials and thickness, if measured.

5.5 Qualification based on standard test joint specimen

5.5.1 General

The preparation and micro-joining of test specimens should be in accordance with 5.4.2. Fulfilment of the requirements of ISO 17279-2 can serve to qualify the personnel performing micro-joining and oxygenation annealing, and testing the standard test joint specimens.

5.5.2 Test specimens

The length and number of test specimens shall be sufficient to allow all required tests to be performed. Specimens shall have dimensions illustrated in Figure 4 or Figure 5, and at least 3 test specimens shall be required for the test, respectively.

Perfect removal of Cu stabilizer and or Ag overlayer is critically important not to contaminate the superconducting layers during micro-joining. Elimination procedure of these materials shall be established and Cu stabilizer and or Ag overlayer shall be completely removed by the established procedure before micro-joining. The 2G ReBCO HTS virgin material and faying surfaces shall be sufficiently free of surface oxides, protective finishes, adhesives, oils, grease, dirt, and any other contaminants that can have a detrimental effect on joint quality.

The specimen identifications shall be marked on the test specimen before the test.

5.5.3 Micro-joining and oxygenation-annealing procedure of test specimens

5.5.3.1 General

The procedures described in this document are examples and recommended procedures. Manufacturer(s) or service operator(s) shall establish the procedures depending on design and operation manual of equipment.

The test specimens shall be micro-joined and oxygenation-annealed by welding personnel qualified according to ISO 17279-2 and the WPS. Micro-joining and oxygenation-annealing of the test specimens shall be witnessed by an examiner or other designated personnel. The recommended procedures for micro-joining and oxygenation-annealing of the test specimens are specified in 5.4, 5.5.3.2 and 5.5.3.3, respectively. Annex A shows the procedure check lists.

5.5.3.2 Micro-joining procedure

a) Exposed two superconducting layers are faced each other as shown in Figure 4 and Figure 5.

b) Align the a) specimen on the holder of joining chamber.

c) Set the chamber vacuum level, time to reach the peak temperature (heating rate), peak temperature (maximum joining temperature), pressure to pressurize the b) holder, and dwell time at the peak temperature (joining time).

d) Micro-joining is started by pressurizing the holder (the area to be joined) with pre-set pressure when pre-set peak temperature is reached within pre-set time under pre-set vacuum level.

   — Heating rate should be as high as possible to protect oxygen out-diffusion from the superconducting layers of 2G HTSs.

   — Peak temperature (maximum joining temperature) should be as low as possible to protect oxygen out-diffusion from the superconducting layers of 2G HTSs and to avoid formation of Re_2BaCuO_5, $BaCuO_2$, and CuO which provide negative effect on superconductivity through the chemical reaction of $ReBa_2Cu_3O_{7-x} \rightarrow 1/2\ [Re_2BaCuO_5 + (3BaCuO_2 + 2CuO) + O_2]$.

e) Micro-joining is finished when pre-set dwell time at the peak temperature is reached. Holder pressure is back to "0".

f) Supply oxygen so that the vacuum chamber reaches to the atmospheric pressure, and continue to supply oxygen to cool down the chamber and joined superconductor to room temperature. Moderate-fast cooling rate is necessary to stop the oxygen out-diffusion.

g) Pick-up the joined specimen and move it to the oxygenation-annealing chamber.

NOTE 1 Heating rate and cooling rate are automatically determined once temperature and time are set, if the equipment is designed as automatic control.

NOTE 2 Controllers and gauges in the joining chamber can be as follows for example, depending on joining chamber design:

- vcuum controller and gauge, time controller and gauge, temperature controller and gauge;
- current meter, voltage meter and pressure controller and gauge;
- PID[1] controller or any other controller and TPR[2] or any other regulator.

5.5.3.3 Oxygenation-annealing procedure

a) Align the joined 2G HTS obtained through 5.5.3.2 to the oxygenation-annealing chamber as much as possible depending on chamber design. Set the time to reach the oxygenation-annealing temperature from room temperature (heating rate), oxygenation-annealing temperature, oxygen flow rate in the oxygen tank, and dwell time at the oxygenation-annealing temperature (oxygenation-annealing time), time to cool down to room temperature.

b) Heat the superconductor from room temperature to oxygenation-annealing temperature with a pre-determined moderate heating rate.

c) Anneal at predetermined temperature with oxygen supply. Hours or days of oxygenation-annealing are necessary depending on the furnace capacity, oxygen environment, purity and concentration, joint types, etc.

d) Supply uniform oxygen flow into the chamber and release the oxygen uniformly to the atmosphere during entire oxygenation-annealing. Micro oxygen gas release knob is installed in the chamber to control the oxygen release. Control the micro oxygen gas release knob by regulating the oxygen supplying pressure and chamber internal pressure.

e) Check the chamber internal pressure all the time during entire oxygenation-annealing. The chamber internal pressure is the difference between oxygen supplying pressure and oxygen release pressure.

f) Oxygenation annealing is finished at the pre-determined time set.

g) Cool down to room temperature at predetermined cooling rate. Slow cooling rate is required to provide atoms re-arrangement.

NOTE 1 Controllers and gauges in the oxygenation-annealing chamber can be as follows for example, depending on oxygenation-annealing chamber design:

- time controller and gauge, and temperature controller and gauge;
- current meter and voltage meter;
- pressure gauge;
- PID controller or any other controller and TPR or any other regulator.

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[1] PID (Proportional-Integral-Derivative) controller: Once time and temperature are set (Heating rate and Cooling rate), the thermocouple sensor connected to the PID controller checks the holder temperature and PID controller sends signal to the TPR (Thyristor Power Regulator) to adjust current.

[2] TPR (Thyristor Power Regulator): TPR receives the signal from the PID controller and adjusts current to get pre-determined heat input (power). And the TPR sends the current to the heater to get pre-determined temperature.

NOTE 2 Controllers and gauges in the oxygen tank: oxygen gas regulator.

5.5.3.4 Micro-joining and oxygenation-annealing procedure in the same chamber and with continuous operation

a) Exposed two superconducting layers are faced each other as shown in Figure 4 and Figure 5.

b) Align the a) specimen on the holder of joining chamber.

c) Set the chamber vacuum level, time to reach the peak temperature (heating rate), peak temperature (maximum joining temperature), pressure to pressurize the b) holder, and dwell time at the peak temperature (joining time).

d) Micro-joining is started by pressurizing the holder (the area to be joined) with pre-set pressure when pre-set peak temperature is reached within pre-set time under pre-set vacuum level.

- Heating rate should be as high as possible to protect oxygen out-diffusion from the superconducting layers of 2G HTSs.
- Peak temperature (maximum joining temperature) should be as low as possible to protect oxygen out-diffusion from the superconducting layers of 2G HTSs and to avoid formation of Re_2BaCuO_5, $BaCuO_2$, and CuO which provide negative effect on superconductivity through the chemical reaction of $ReBa_2Cu_3O_{7-x} \rightarrow 1/2[Re_2BaCuO_5 + (3BaCuO_2 + 2CuO) + O_2]$.

e) Micro-joining is finished when pre-set dwell time at the peak temperature is reached. Holder pressure is back to "0".

f) Supply oxygen so that the vacuum chamber reaches to the atmospheric pressure, and continue to supply oxygen to cool down the chamber and joined superconductor to oxygenation-annealing temperature. Moderate-fast cooling rate is necessary to stop the oxygen out-diffusion.

NOTE 1 Heating rate and cooling rate are automatically determined once temperature and time are set, if the equipment is designed as automatic control.

NOTE 2 Controllers and gauges in the joining chamber can be as follows for example, depending on joining chamber design:

- vacuum controller and gauge, time controller and gauge, and temperature controller and gauge;
- current meter and voltage meter, and pressure controller and gauge;
- PID[3] controller or any other controller, and TPR[4] or any other regulator.

g) Anneal at predetermined temperature with oxygen supply during hours or days predetermined depending on furnace capacity, oxygen environment, purity and concentration, joint types, and so on.

h) Supply uniform oxygen flow into the chamber and release the oxygen uniformly to the atmosphere during entire oxygenation-annealing. Micro oxygen gas release knob is installed in the chamber to control the oxygen release. Control the micro oxygen gas release knob by regulating the oxygen supplying pressure and chamber internal pressure.

i) Check the chamber internal pressure all the time during entire oxygenation-annealing. The chamber internal pressure is the difference between oxygen supplying pressure and oxygen release pressure.

j) Oxygenation annealing is finished at the pre-determined time set.

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[3] PID (Proportional-Integral-Derivative) controller: Once time and temperature are set (Heating rate and Cooling rate), the thermocouple sensor connected to the PID controller checks the holder temperature and PID controller sends signal to the TPR (Thyristor Power Regulator) to adjust current.

[4] TPR (Thyristor Power Regulator): TPR receives the signal from the PID controller and adjusts current to get pre-determined heat input (power). And the TPR sends the current to the heater to get pre-determined temperature.

k) Cool down to room temperature. Slow cooling rate is required to provide atoms re-arrangement.

l) Pick-up the joined and oxygenation-annealed specimen.

NOTE 3 Controllers and gauges in the oxygenation-annealing chamber can be as follows for example, depending on oxygenation-annealing chamber design:

- time controller and gauge, and temperature controller and gauge;
- current meter, voltage meter, and pressure gauge;
- PID controller or any other controller, and TPR or any other regulator.

NOTE 4 Controllers and gauges in the oxygen tank: oxygen gas regulator.

5.5.4 Testing of test specimens

5.5.4.1 General

For testing the micro-joined and oxygenation-annealed specimens, see ISO 17279-3.[5)] Personnel performing micro-joining and oxygenation annealing, and testing the test specimens shall be qualified in accordance with ISO 17279-2, or equivalent. The acceptance criteria are in 5.9. Extent of testing and test methods are described in 5.5.4.2 to 5.5.4.8. 5.5.4.9 describes re-test requirement. Micro-joining and oxygenation annealing, and testing of the test specimens shall be witnessed by an inspector or designated personnel.

5.5.4.2 Extent of testing

Table 1 shows procedure qualification test requirements for micro-joining and oxygenation-annealing. Specific service or manufacturing conditions can require more comprehensive testing in order to obtain additional test data.

Table 1 — Purpose of testing and test requirements

Type of testing	Extent of testing	Confirmation of testing	Procedure qualification test required
Visual testing	3 joined specimens	Verify the joint is free from any imperfections and de-bonding, etc., and check appearances and alignments, etc.	Yes
Four-point-probes testing	3 joined specimens (3 specimens cover I_c, J_c, and n-value)	Verify the joint critical current, I_c, is 80 % and higher to the virgin materials of 2G HTS.	Yes
		Verify the joint critical current density, J_c, is 40 % and higher to the virgin materials of 2G HTS. NOTE The joint cross-sectional area of superconducting layer is twice of that of virgin material.	No[a]
		Verify the joint n-value is 80 % and higher to the virgin materials of 2G HTS.	No[a]
	According to manufacturer's requirements	Verify the joint cyclic (at least 5 times, or more than 5 times if required by manufacturer) thermal shock characteristic.	No[a]

[a] If required by manufacturer, it shall be yes.

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5) Under preparation.

Table 1 *(continued)*

Type of testing		Extent of testing	Confirmation of testing	Procedure qualification test required
Field-decay testing		1 joined specimen from the qualified I_c joined test specimen	Verify the joint electric resistance and magnetic field strength variation over time	No[a]
In-field testing			to verify the joint I_c or J_c variations at different cryogenic conditions (20 K, 30 K, 50 K, and 77 K) under different externally applied magnetic field (0 T, 0,35 T, 0,7 T, 1,0 T, 2,0 T, and 3,0 T, where T is magnetic strength unit in terms of Tesla).	No[a]
Tensile testing	Without reinforcement	3 joined specimens from the qualified I_c joined test specimens	Measure joint tensile strength and I_c profiles during tensile loading as a reference.	No[a]
	With reinforcement		Verify joint tensile strength and I_c are equal to the virgin materials of 2G HTS.	Yes
Bend testing	Without reinforcement	3 joined specimens from the qualified I_c joined test specimen	Measure joint bend diameter and I_c profiles during bending load as a reference.	No[a]
	With reinforcement	1 joined specimen from the qualified I_c joined test specimen	Determine joint bend diameter to show the same I_c as virgin materials of 2G HTS.	No[a]
Critical magnetic field testing		1 joined specimen from the qualified I_c joined test specimen	Verify the joint critical magnetic field (B_c) characteristic.	No[a]
Critical current density distribution testing	Magneto-optical technique or hall sensor scanning technique	1 joined specimen from the qualified I_c joined test specimen	Verify the joint critical current density distribution profiles and characteristics.	No[a]
Microscopes or X-ray diffraction testing	OM, SEM, TEM, XRD	1 joined specimen from the qualified I_c joined test specimen per each test	Verify the characteristics of joint materials.	No[a]

[a] If required by manufacturer, it shall be yes.

5.5.4.3 Visual testing

The joints of test specimens shall be visually tested after micro-joining and oxygenation-annealing. The joint shall be especially checked for appearances, imperfections, alignments, de-bonding, etc.

5.5.4.4 Four-point-probes testing

The self-field critical current, I_c, values of the 2G HTS joint shall be measured in a bath of liquid nitrogen (LN2) or other media using a 1 µV/cm criterion from the voltage (V)–current (I) curve obtained by 4-point-probes technique. Joint critical current, I_c, value is extremely important to confirm joint integrity. From this testing, critical current density, J_c, n-value, and cyclic thermal shock characteristic can also be determined.

J_c characterizes the maximum electrical transport current per cross-sectional unit area that the superconductor is able to maintain without resistance and is expressed in terms of I_c divided by joined cross-sectional area. N-value characterizes the sharpness of a resistive transition in the V–I curve at I_c measurement. The higher the n-value, the lower the resistivity and the better superconductivity. The cyclic thermal shock is to determine the I_c variations or profiles of the joint in cyclic thermal shock environments, which is at least 5 times measurements or more than 5 times if required by manufacturer in liquid nitrogen bath.

5.5.4.5 Field decay testing

To determine the resistance of a 2G HTS closed loop with a 2G HTS joint to a level below 10^{-12} Ω and magnetic field strength variation over time, a field-decay technique is used.

5.5.4.6 In-field testing

I_c or J_c may be tested at 20 K, 30 K, 50 K, and 77 K cryogenic conditions under externally applied magnetic field of 0 T, 0,35 T, 0,7 T, 1,0 T, 2,0 T, and 3,0 T at perpendicular or parallel angle or different angles to the magnetic field according to the manufacturer's requirement. Other cryogenic conditions under other externally applied magnetic fields may be applied depending on the manufacturer's requirement.

5.5.4.7 Tensile testing

Each joined specimen with or without reinforcement may be tensile tested both at room temperature and cryogenic condition 77 K (−196 °C) or other required temperature. The tests in cryogenic condition can determine the critical tensile values which show I_c. The joint shall be reinforced all around the joint firmly by epoxy coating or other means when required.

5.5.4.8 Bend testing

The joined specimen with or without reinforcement may be bent according to the manufacturer's requirements. Each joined specimen may be bend tested both at room temperature and cryogenic condition 77 K (−196 °C) or other required temperature. The tests in cryogenic condition can determine the critical bend diameter in both tensile and compression which show I_c. The joint shall be reinforced all around the joint firmly by epoxy coating or other means when required.

5.5.4.9 Critical magnetic field testing

Critical magnetic field (B_c) may be tested at 20 K, 30 K, 50 K, and 77 K cryogenic conditions at perpendicular or parallel angle or different angles to the magnetic field. Other cryogenic conditions under other externally applied magnetic fields may be applied depending on the manufacturer's requirement.

5.5.4.10 Critical current density distribution testing

The technique for the non-destructive evaluation of the critical current density, J_c, distribution is to measure magnetic field distribution generated by the J_c distribution at the joint. The magnetic field due to J_c distribution can be measured by a magneto-optical technique or by a Hall sensor scanning technique.

5.5.4.11 Microscopes and X-ray diffraction testing

The joint may be examined by optical microscope (OM), scanning electron microscope (SEM), transmission electron microscope (TEM), or X-ray diffraction (XRD) to characterize the joint materials.

OM examines surface features and characteristics of microstructures, grains and grain boundaries of the joint. SEM detects the composition of the unknown materials of the joint and the joint surface topography and TEM can image and analyze the structure of materials using a high energy electron beam transmitted through a very thin sample (<50 nm).

XRD measures the average spacing between layers or rows of atoms, determines the orientation of a single crystal or grain, finds the crystal structure of an unknown material, and measures the size, shape and internal stress of small crystalline regions in the joint.

5.5.5 Re-testing

If one of three specimens fails to meet the acceptance criteria of 5.9, the test shall be rejected. Two additional test specimens shall be micro-joined and oxygenation-annealed using the same procedure and subjected to testing. If one of these extra specimens fails to meet the requirements, additional two procedure qualification tests for micro-joining and oxygenation-annealing shall be required. If two of three specimens or all three specimens fail to meet the acceptance criteria of 5.8, the procedure re-qualification according to 5.5 shall be required.

5.5.6 Test record

The results of all testing required in Table 1 of 5.5.4.2 and tests required by manufacturer shall be documented. The format of the documentation shall be decided by the manufacturer. A suggested test record is shown in Annex C. The documentation can be on paper or electronic media.

5.6 Range of qualification

5.6.1 General

Each of the conditions given in 5.4 and 5.5 shall be met. Additions, deletions or changes outside the optimized and specified shall require a new micro-joining and oxygenation-annealing procedure qualification test to be performed.

5.6.2 Related to the manufacturer

Micro-joining and oxygenation-annealing procedure qualification tests carried out by a manufacturer in workshops, sites or laboratories under the technical and quality control of manufacturer are valid. Micro-joining and oxygenation-annealing procedure qualification tests carried out by other professional test organizations in workshops, sites or laboratories under the same technical and quality control of manufacturer are also valid.

5.6.3 Essential variables

The qualification of WPS is based on essential variable parameters. For each essential variable parameter, optimized conditions are defined. If a micro-joining and or oxygenation-annealing is required outside the range of qualification, a new qualification test is required. A successful joint qualification test made by any specific method qualifies only for that micro-joining and oxygenation-annealing method. The total joint thickness for 2G HTS superconducting materials is only between 2 µm and 4 µm thick depending on pressure during joining. The micro-joining methods of superconducting layers include a) pressurized partial micro-melting diffusion, or b) pressurized solid-state diffusion, or both. The micro-joining and oxygenation-annealing apparatuses include, but are not limited to, mechanized and semi-automated joining equipment.

The essential variables during micro-joining under vacuum environment and oxygenation annealing under pressurized oxygen environment are extremely sensitive to the joint qualification. The variables include joint design, joint width, joint materials, micro-joining and oxygenation-annealing apparatuses, chamber vacuum level, heating rate, peak temperature for the joining, pressure to the joint, dwell time at the peak temperature, cooling rate down to room temperature or oxygenation-annealing temperature, heating rate to oxygenation-annealing temperature, oxygenation-annealing temperature, dwell time at the oxygenation-annealing temperature, oxygen flow rate during oxygenation-annealing, oxygen pressure to the joint, cooling rate down to room temperature. Any changes of essential variables optimized require re-qualification.

Table 2 shows essential variables of joint design, joint widths, superconducting materials, and apparatuses. Table 3 shows essential variable parameters for micro-joining and oxygenation-annealing. These parameters are critical to the joint quality.

Table 2 — Essential variables required for optimization of joint, materials and apparatuses

Joint design		Joint width (mm)	Superconducting materials	Micro-joining and oxygenation-annealing chambers (furnaces)
Lap	Bridge	3 mm, 4 mm, 6 mm, 12 mm depending on specifications	YBCO, GdBCO, SmBCO, or others	Design details and functions of each controller

Table 3 — Essential variables required for optimization of micro-joining and oxygenation-annealing parameters

Micro-joining parameters	Oxygenation- annealing parameters
Removal of Cu stabilizer and or Ag overlayer	Heating rate
Chamber internal vacuum level	Annealing Temperature
Heating rate	Dwell time
Joining peak temperature	Oxygen flow rate
Pressure to the joint	Chamber internal pressure
Dwell time at the joining peak temperature	Cooling rate to room temperature
Cooling rate to room temperature or oxygenation-annealing temperature	

5.6.4 Other variables

The range of qualification for other variables (i.e. surface roughness of superconducting layer) may be specified in the WPS.

5.7 Micro-joining and oxygenation-annealing procedure specification and procedure qualification record

The welding procedure qualification record (WPQR) is a statement of the results of assessing each joined test specimen, including re-tests. The relevant items listed in the WPS shall be included, together with details of any features that would be tested. For test methods, see ISO 17279-3[6] and evaluated in accordance with 5.9. If the test results are acceptable, the WPQR is qualified and shall be signed, dated and issued by an examiner or designated personnel. Welding procedure specification (WPS) is qualified and shall be issued when WPQT is qualified and WPQR is issued.

Examples of checklist for micro-joining and oxygenation-annealing, pWPS for the procedure qualification test, WPQR form and WPS are shown in Annex A, B, C and D, respectively.

5.8 Final treatment of production run joints

All joints produced with qualified and certified procedure according to WPS in the production run shall be firmly reinforced all around the joint to protect de-bonding and eliminate notches and associated stress concentration of the joints against external forces and stresses during handling or operation. The reinforcement shall be done after completing the whole process including oxygenation annealing, and may apply after four-point-probes testing to confirm critical current, I_c.

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[6] Under preparation.

5.9 Acceptance criteria

The test specimens or production-run joints shall meet the appropriate acceptance criteria specified in Table 4 to Table 12.

Table 4 — Visual testing

Test items	Acceptance criteria
Appearances, imperfections, alignments, etc.	Accept when manufacturer's requirement are met.
De-bonding	Reject
Slight tensioning in the direction of length by pulling the joined 2G HTS with two fingers of both hands	Reject when separated

Table 5 — Four-point-probes testing

Test items	Acceptance criteria
Self-field critical current (I_c) value	80 % of virgin 2G ReBCO HTS or manufacturer's specification. The same I_c as virgin 2G HTS may require in NMR (Nuclear Magnetic Resonance) or MRI (Magnetic Resonance Imaging) magnet manufacturing
Critical current density (J_c) value	40 % of virgin 2G ReBCO HTS or manufacturer's specification
n-value	80 % of virgin 2G ReBCO HTS or manufacturer's specification
Self-field critical current (I_c) profiles by cyclic thermal shock	80 % of virgin 2G ReBCO HTS or manufacturer's specification. The same I_c as virgin 2G HTS may require in NMR or MRI magnet manufacturing

Table 6 — Field decay testing

Test items	Acceptance criteria
Joint resistance	10^{-15} Ω
Induced magnetic field at certain time (30 d, 60 d, 90 d, 120 d, 180 d, 240 d, 365 d, ---)	Manufacturer's specification, or ($B(t)/B(t_0)$) > 0,98 at 120 d Where, $B(t)$ = Induced magnetic field (Tesla) at time t and $B(t_0)$ = Initial magnetic field (Tesla) at time "0"

Table 7 — In-field testing

Test items	Acceptance criteria
Self-field critical current (I_c) value	Manufacturer's specification

Table 8 — Tensile testing

Temperature	Test items	Acceptance criteria
Room temperature	Joined bare specimen	Manufacturer's specification or 20 % of virgin parent sample
	Joined and reinforced specimen	Manufacturer's specification or 100 % of virgin parent sample
77 K Cryogenic	Joined bare specimen	Manufacturer's specification or 20 % of virgin parent sample
	Joined and reinforced specimen	Manufacturer's specification or 100 % of virgin parent sample (Critical tensile value)
Other temperature	Joined bare specimen	Manufacturer's specification
	Joined and reinforced specimen	Manufacturer's specification

Table 9 — Bend testing

No	Test items	Acceptance criteria
Room temperature	Joined bare specimen	Manufacturer's specification
	Joined and reinforced specimen	Manufacturer's specification or Bend diameter 11 mm and over
77 K Cryogenic	Joined bare specimen	Manufacturer's specification
	Joined and reinforced specimen	Manufacturer's specification or Critical bend diameter 11 mm and over
Other temperature	Joined bare specimen	Manufacturer's specification
	Joined and reinforced specimen	Manufacturer's specification

Table 10 — Critical magnetic field testing

Test items	Acceptance criteria
Critical magnetic field, B_c, values at different cryogenic conditions at perpendicular or parallel angle or different angles to the magnetic field	Manufacturer's specification

Table 11 — Critical current density distribution testing

Test items	Acceptance criteria
Distribution profiles of critical current density (J_c)	Manufacturer's specification

Table 12 — OM, SEM, TEM or XRD testing

Test items	Acceptance criteria
Materials characteristics	Manufacturer's specification

5.10 Identification and traceability

Identification and traceability of a joint to a WPS and personnel for joining and testing shall be maintained throughout the manufacturing process.

6 Third-party check

If there are any findings during reviewing and monitoring, the findings can be directly notified to the president or responsible personnel in the organization by issuing an NCR (non-conformance report). An informative check list is in Annex E.

Annex A
(informative)

Micro-joining and oxygenation-annealing procedure

Micro-joining procedure in joining chamber			
No.	Items	Check	Remarks
1	Exposed two superconducting layers are faced each other		
2	Align the specimen set on the holder of joining chamber		
3	Set the chamber vacuum level, time to reach the peak temperature (heating rate), peak temperature (maximum joining temperature), pressure to pressurize the holder, and dwell time at the peak temperature (joining time)		
4	Micro-joining is started by pressurizing the holder (the area to be joined) with pre-set pressure when pre-set peak temperature is reached within pre-set time under pre-set vacuum level. — Heating rate should be as high as possible to protect oxygen out-diffusion from the superconducting layers. — Peak temperature (maximum joining temperature) should be as low as possible to protect oxygen out-diffusion from the superconducting layers and to avoid formation of Gd_2BaCuO_5, $BaCuO_2$, and CuO which provide detrimental effect on superconductivity through the chemical reaction of $GdBa_2Cu_3O_{7-x} \rightarrow 1/2[Gd_2BaCuO_5 + (3BaCuO_2 + 2CuO) + O_2]$		
5	Micro-joining is finished when pre-set dwell time at the peak temperature is reached. Holder pressure is back to "0"		
6	Supply oxygen so that the vacuum chamber reaches to the atmospheric pressure, and continue to supply oxygen to cool down the chamber and joined superconductor to room temperature. Moderate-fast cooling rate is necessary to stop the oxygen out-diffusion		
7	Pick-up the joined specimen and move it to the oxygenation-annealing chamber		
Additional check items			
Heating rate and cooling rate are automatically determined once temperature and time are set.			
Controllers and gauges in the joining chamber			
Vacuum controller and gauge			
Time controller and gauge			
Temperature controller and gauge			
Current meter			

Voltage meter		
Pressure controller and gauge PID (Proportional-Integral-Derivative) controller or any other controller Once time and temperature are set (Heating rate and Cooling rate), the thermocouple sensor connected to the PID controller checks the holder temperature and PID controller sends signal to the TPR (Thyristor Power Regulator) to adjust current		
TPR (Thyristor Power Regulator) or any other regulator TPR receives the signal from the PID controller and adjust current to get pre-determined heat input (power). And the TPR sends the current to the heater (halogen lamp or whatever) to get pre-determined temperature		

Oxygenation-annealing procedure in oxygenation-annealing chamber			
No.	Items	Check	Remarks
1	Align the joined superconductor obtained through Joining process in joining chamber to the tube in the chamber as many as possible depending on chamber design		
2	Set the time to reach the oxygenation annealing temperature from room temperature (heating rate), oxygenation annealing temperature, oxygen flow rate in the oxygen tank, and dwell time at the oxygenation annealing temperature (oxygenation-annealing time), time to cool down to room temperature		
3	Heat the superconductor from room temperature to oxygenation-annealing temperature with moderate heating rate predetermined		
4	Anneal at predetermined temperature (just under phase transition temperature from superconducting orthorhombic phase to a non-superconducting tetragonal phase) with oxygen supply. Hours or days of oxygenation-annealing are necessary depending on furnace capacity, oxygen environment, purity and concentration, joint types, and so on.		
5	Supply uniform oxygen flow into the chamber and release the oxygen uniformly to the atmosphere during entire oxygenation annealing. Micro oxygen gas release knob is installed in the chamber to control the oxygen release. Control the micro oxygen gas release knob by regulating the oxygen supplying pressure and chamber internal pressure		
6	Check the chamber internal pressure all the time during entire oxygenation-annealing. The chamber internal pressure is the difference between oxygen supplying pressure and oxygen release pressure		
7	Oxygenation-annealing is finished at the pre-determined time set		
8	Cool down to room temperature at predetermined cooling rate. Slow cooling rate is required to provide atoms re-arrangement		
Additional check items			
Controllers and gauges in the oxygenation annealing chamber			
Time controller and gauge			
Temperature controller and gauge			
Current meter			
Voltage meter			

Pressure controller and gauge		
PID (Proportional-Integral-Derivative) or any other controller		
TPR (Thyristor Power Regulator) or any other reglator		

Micro-joining and oxygenation-annealing procedure in oxygenation-annealing chamber and with continuous operation			
No.	Items	Check	Remarks
1	Exposed two superconducting layers are faced each other		
2	Align the specimen set on the holder of joining chamber		
3	Set the chamber vacuum level, time to reach the peak temperature (heating rate), peak temperature (maximum joining temperature), pressure to pressurize the holder, and dwell time at the peak temperature (joining time)		
4	Micro-joining is started by pressurizing the holder (the area to be joined) with pre-set pressure when pre-set peak temperature is reached within pre-set time under pre-set vacuum level. — Heating rate should be as high as possible to protect oxygen out-diffusion from the superconducting layers. — Peak temperature (maximum joining temperature) should be as low as possible to protect oxygen out-diffusion from the superconducting layers and to avoid formation of Gd_2BaCuO_5, $BaCuO_2$, and CuO which provide detrimental effect on superconductivity through the chemical reaction of $GdBa_2Cu_3O_{7-x} \rightarrow 1/2[Gd_2BaCuO_5 + (3BaCuO_2 + 2CuO) + O_2]$		
5	Micro-joining is finished when pre-set dwell time at the peak temperature is reached. Holder pressure is back to "0"		
6	Supply oxygen so that the vacuum chamber reaches to the atmospheric pressure, and continue to supply oxygen to cool down the chamber and joined superconductor to room temperature. Moderate-fast cooling rate is necessary to stop the oxygen out-diffusion		
7	Pick-up the joined specimen and move it to the oxygenation-annealing chamber		
Additional check items during joining			
Heating rate and cooling rate are automatically determined once temperature and time are set.			
Controllers and gauges in the joining chamber			
Vacuum controller and gauge			
Time controller and gauge			
Temperature controller and gauge			
Current meter			
Voltage meter			
Pressure controller and gauge PID (Proportional-Integral-Derivative) controller or any other controller Once time and temperature are set (Heating rate and Cooling rate), the thermocouple sensor connected to the PID controller checks the holder temperature and PID controller sends signal to the TPR (Thyristor Power Regulator) to adjust current			

TPR (Thyristor Power Regulator) or any other regulator TPR receives the signal from the PID controller and adjust current to get pre-determined heat input (power). And the TPR sends the current to the heater (halogen lamp or whatever) to get pre-determined temperature			
8	Anneal at predetermined temperature (just under phase transition temperature from superconducting orthorhombic phase to a non-superconducting tetragonal phase) with oxygen supply during hours or days predetermined depending on furnace capacity, oxygen environment, purity and concentration, joint types, and so on.		
9	Supply uniform oxygen flow into the chamber and release the oxygen uniformly to the atmosphere during entire oxygenation-annealing. Micro oxygen gas release knob is installed in the chamber to control the oxygen release. Control the micro oxygen gas release knob by regulating the oxygen supplying pressure and chamber internal pressure.		
10	Check the chamber internal pressure all the time during entire oxygenation-annealing. The chamber internal pressure is the difference between oxygen supplying pressure and oxygen release pressure		
11	Oxygenation-annealing is finished at the pre-determined time set		
12	Cool down to room temperature at predetermined cooling rate. Slow cooling rate is required to provide atoms re-arrangement		
13	Pick-up the joined and oxygenation-annealed specimen		
Additional check items			
Controllers and gauges in the oxygenation annealing chamber			
Time controller and gauge			
Temperature controller and gauge			
Current meter			
Voltage meter			
Pressure controller and gauge			
PID (Proportional-Integral-Derivative) or any other controller			
TPR (Thyristor Power Regulator) or any other regulator			

Annex B
(informative)

Preliminary Welding Procedure Specification (pWPS)

Manufacturer's pWPS No.:____

Joining operator's name:____

2G HTS superconducting material type (YBCO, GdBCO, SmBCO, etc.):____________________

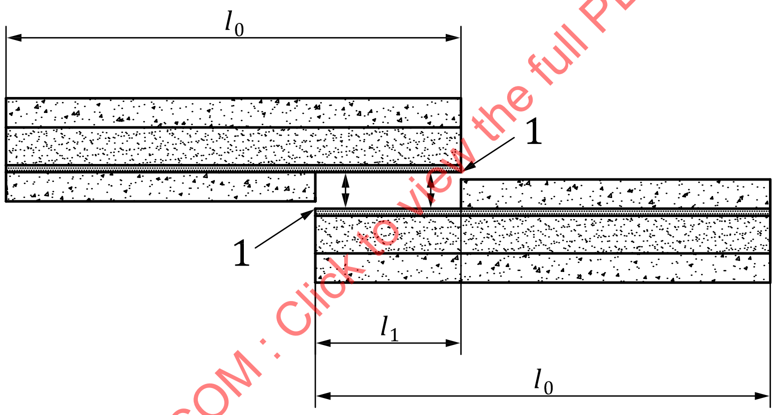
Joint width (mm):____

Superconducting layer thickness (µm):______________________________

Equipment identification (model, serial number, and manufacturer):____________________

Joint preparation (Cu and or Ag layer removal):____________________________

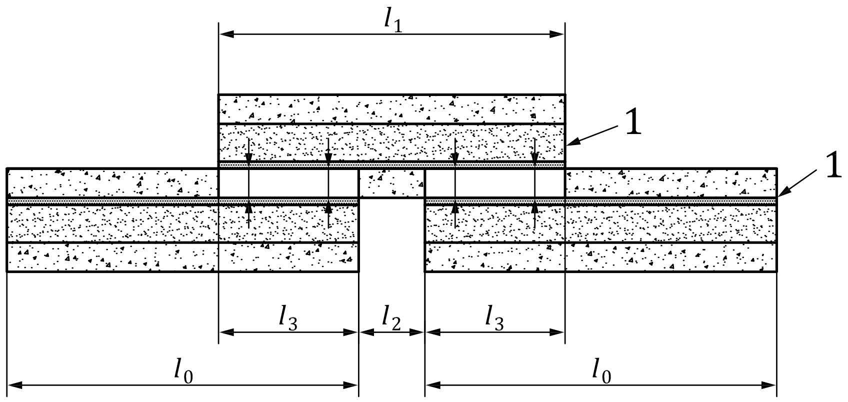
Test specimen arrangement for joint, lap or bridge, and dimensions (sketch):



Key

1	superconducting layer
l_0	60 mm
l_1	40 mm

Figure B.1 — Lap joint



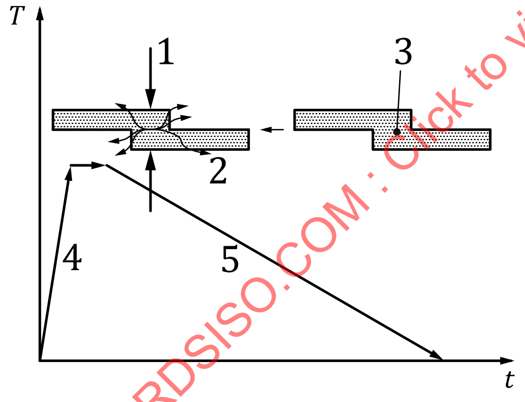
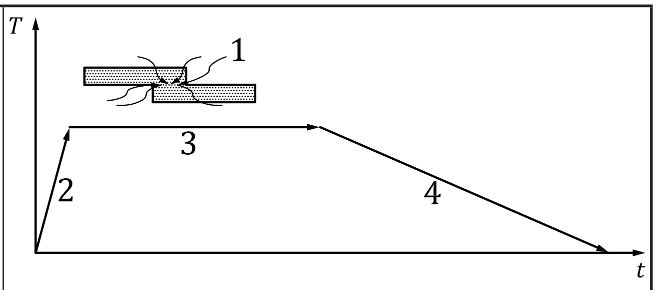
Key

1	superconducting layer
l_0	50 mm
l_1	40 mm to 50 mm
l_2	0 mm to 10 mm
l_3	20 mm

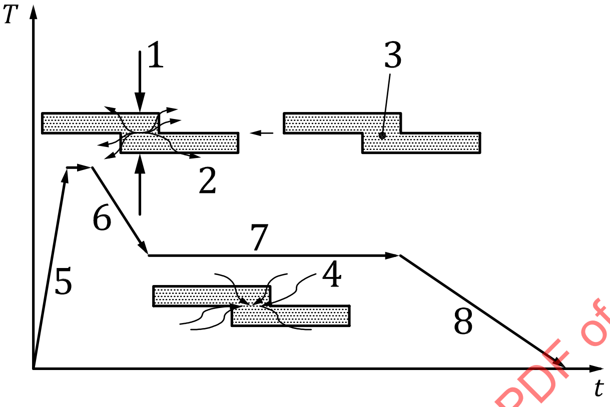
Figure B.2 — Bridge joint

Micro-joining and oxygenation-annealing

Heat treatment cycle (sketch) and data

 NOTE Heat and pressure under vacuum during joining. **Key** 1 heat and pressure 2 oxygen out-diffusion 3 peel-off stabilizers and/or overlayers 4 fast heating 5 fast-moderate cooling	 **Key** 1 oxygen in-diffusion 2 moderate heating 3 oxygenation annealing 4 slow cooling

Vacuum (kPa): Heating rate (°C/s): Peak temperature (°C): Pressure to the joint (MPa): Dwell time at peak temperature (s, or min): Cooling rate down to room temperature (°C/s): Other specifics:	Heating rate (°C/s): Oxygenation annealing temperature (°C): Oxygen flow rate and purity (cm^3/min and %): Chamber pressure (kPa): Dwell time at oxygenation annealing temperature (h): Cooling rate down to room temperature (°C/s): Other specifics:



NOTE Heat and pressure under vacuum during joining.

Key

1	heat and pressure	5	fast heating
2	oxygen out-diffusion	6	fast-moderate cooling
3	peel-off stabilizers and/or overlayers	7	oxygenation annealing
4	oxygen in-diffusion	8	slow cooling

Vacuum (kPa): Heating rate (°C/s): Peak temperature (°C): Pressure to the joint (MPa): Dwell time at peak temperature (s, or min): Cooling rate to oxygenation annealing temperature (°C/s): Other specifics:	Oxygenation annealing temperature (°C): Oxygen flow rate and purity (cm^3/min and %): Chamber pressure (kPa): Dwell time at oxygenation annealing temperature (h): Cooling rate down to room temperature (°C/s): Other specifics:

Manufacturer:______________________________ Date:___________________________________

Name:_____________________________________ Signature:____________________________

Annex C
(informative)

Procedure qualification for micro-joining and oxygenation annealing and Welding Procedure Qualification Record (WPQR)

Manufacturer:______________________________

Address:______________________________

Manufacturer's pWPS No.:______________________________

Manufacturer's WPQR No.:______________________________

Examiner or examining body:______________________________

Joining operator's name:______________________________

ISO Code/testing standard:______________________________

Date of joining:______________________________

Reference No. (if any):______________________________

2G HTS superconducting material type (YBCO, GdBCO, SmBCO, etc.):____________________

2G HTS virgin material width (mm):______________________________

Superconducting layer thickness:______________________________

Equipment identification (model, serial number, and manufacturer):____________________

____

The signature below certifies that the test specimens were prepared, joined and tested satisfactorily in accordance with the requirements of the ISO code/testing standard indicated above.

Location:______________________________ Date of issue:______________________________

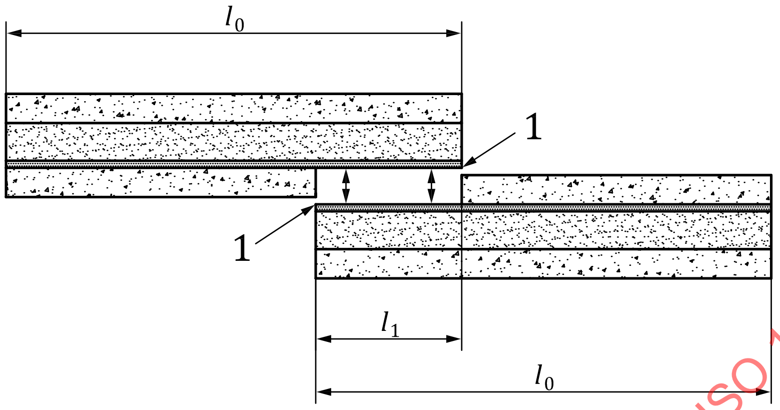
____

Name, date, and signature of examiner or designated personnel

Test results

Joint design

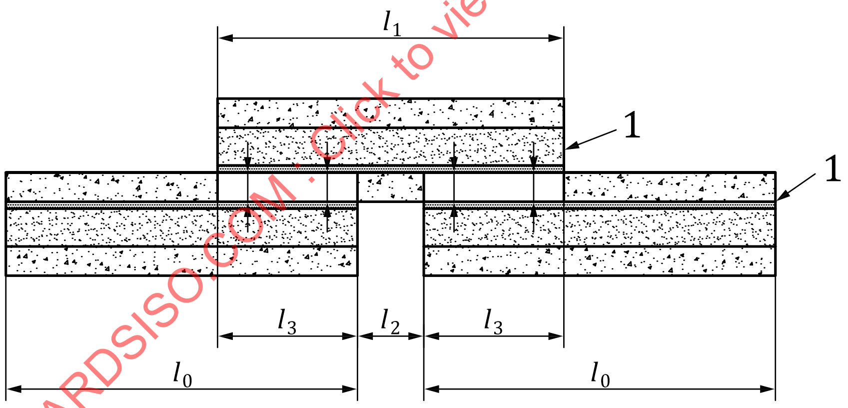
Test specimen arrangement for joint, lap or bridge (sketch):



Key

1	superconducting layer
l_0	60 mm
l_1	40 mm

Figure C.1 — Lap joint



Key

1	superconducting layer
l_0	50 mm
l_1	40 mm to 50 mm
l_2	0 mm to 10 mm
l_3	20 mm

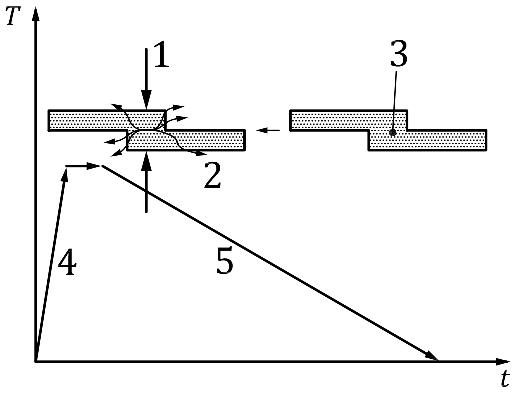
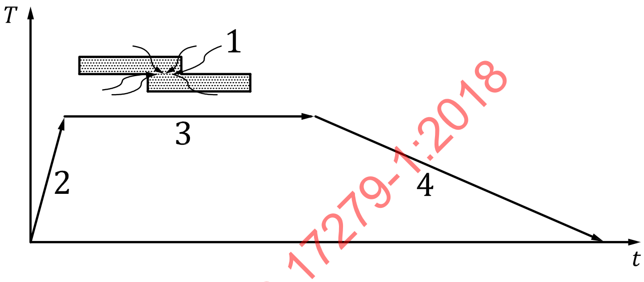
Figure C.2 — Bridge joint

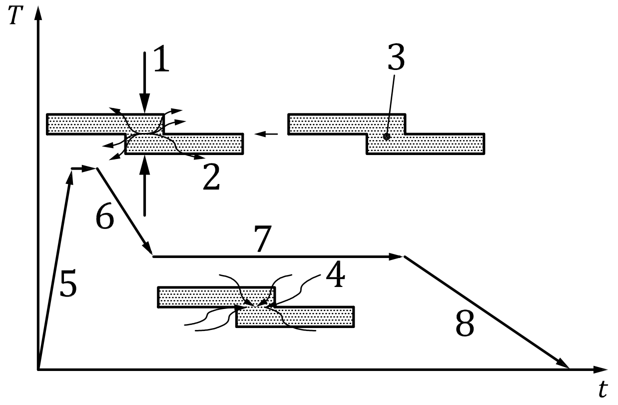
Joint preparation (Cu and or Ag layer removal):____

____

Micro-joining and oxygenation-annealing

Heat treatment cycle (sketch) and data

 NOTE Heat and pressure under vacuum during joining. **Key** 1 heat and pressure 2 oxygen out-diffusion 3 peel-off stabilizers and/or overlayers 4 fast heating 5 fast-moderate cooling	 **Key** 1 oxygen in-diffusion 2 moderate heating 3 oxygenation annealing 4 slow cooling
Vacuum (kPa): Heating rate (°C/s): Peak temperature (°C): Pressure to the joint (MPa): Dwell time at peak temperature (s, or min): Cooling rate down to room temperature (°C/s): Other specifics:	Heating rate (°C/s): Oxygenation annealing temperature (°C): Oxygen flow rate and purity (cm^3/min and %): Chamber pressure (kPa): Dwell time at oxygenation annealing temperature (h): Cooling rate down to room temperature (°C/s): Other specifics:



NOTE Heat and pressure under vacuum during joining.

Key

1	heat and pressure	5	fast heating
2	oxygen out-diffusion	6	fast-moderate cooling
3	peel-off stabilizers and/or overlayers	7	oxygenation annealing
4	oxygen in-diffusion	8	slow cooling

Vacuum (kPa): Heating rate (°C/s): Peak temperature (°C): Pressure to the joint (MPa): Dwell time at peak temperature (s, or min): Cooling rate to oxygenation annealing temperature (°C/s): Other specifics:	Oxygenation annealing temperature (°C): Oxygen flow rate and purity (cm^3/min and %): Chamber pressure (kPa): Dwell time at oxygenation annealing temperature (h): Cooling rate down to room temperature (°C/s): Other specifics:

Results of micro-joining and oxygenation-annealing

Visual testing

Tests carried out in accordance with the requirements of:________________________________

Laboratory report reference No., if any:________________________________

Remarks:________________________________

Test results were acceptable/not acceptable:________________________________

Four-point-probes testing

Tests carried out in accordance with the requirements of:________________________________

Laboratory report reference No., if any:________________________________

Remarks:________________________________

Test results were acceptable/not acceptable:________________________________