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Determination of resistance to intergranular corrosion of stainless steels —

Part 3: Corrosion test for low-Cr ferritic stainless steels

Détermination de la résistance à la corrosion intergranulaire des aciers inoxydables —

Partie 3: Essai de corrosion pour les aciers inoxydables ferritiques à faible teneur en chrome

Reference number
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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

A list of all parts in the ISO 3651 series can be found on the ISO website.

Introduction

The term “intergranular corrosion test” denotes the corrosion test carried out by means of preferential attack of the grain boundaries.

The low-Cr [less than 16 % Cr, less than 0,3 % Ni, less than 0,3 % Ti and less than 0,3 % Nb, less than 0,3 % (Ti + Atomic weight ratio Nb)] ferritic stainless steels may be subject to such attack when they have been exposed to a temperature between about 500 °C to 1 300 °C. This heat cycle, which may provoke sensitization to intergranular corrosion, may occur during rolling or welding operation.

Low-Cr ferritic stainless steels may show high risks to uniform corrosion rates and copper deposit when tested by the methods given by ISO 3651-1 and ISO 3651-2 since the electrochemical potential difference between the matrix and the Cr depletion for low-Cr ferritic stainless steels is much less than that of medium-Cr ferritic stainless steels. These risks should be considered in selecting this test method. Application of this standard test to the other stainless steels out of the above specifications should be made based on the specific agreement between the interested parties.

Determination of resistance to intergranular corrosion of stainless steels —

Part 3:
Corrosion test for low-Cr ferritic stainless steels

1 Scope

This document specifies the determination of the intergranular corrosion susceptibility of low-Cr [less than 16 % Cr, less than 0,3 % Ni, less than 0,3 % Ti and less than 0,3 % Nb, less than 0,3 % (Ti + Atomic weight ratio Nb)] ferritic stainless steels in the 0,5 % sulfuric acid/copper sulfate test. It also specifies the purposes which may be assigned to the test.

The method is applicable to stainless steels supplied in the form of rolled sheets and welded tubes and intended for use in a mildly oxidizing acid medium.

It is important to note that the result of the corrosion test is only strictly valid for the corrosive medium used in the test. It constitutes a basis for estimating the resistance to intergranular corrosion but cannot be used to check resistance to other forms of corrosion (general corrosion, pitting, stress corrosion, etc.). It is important for the user to adapt the specified corrosion test where steels are used. This test is, in no case, considered as an absolute criterion of the quality of the metal.

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 8044, *Corrosion of metals and alloys — Basic terms and definitions*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8044 apply.

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

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

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

4 Preparation of test specimens

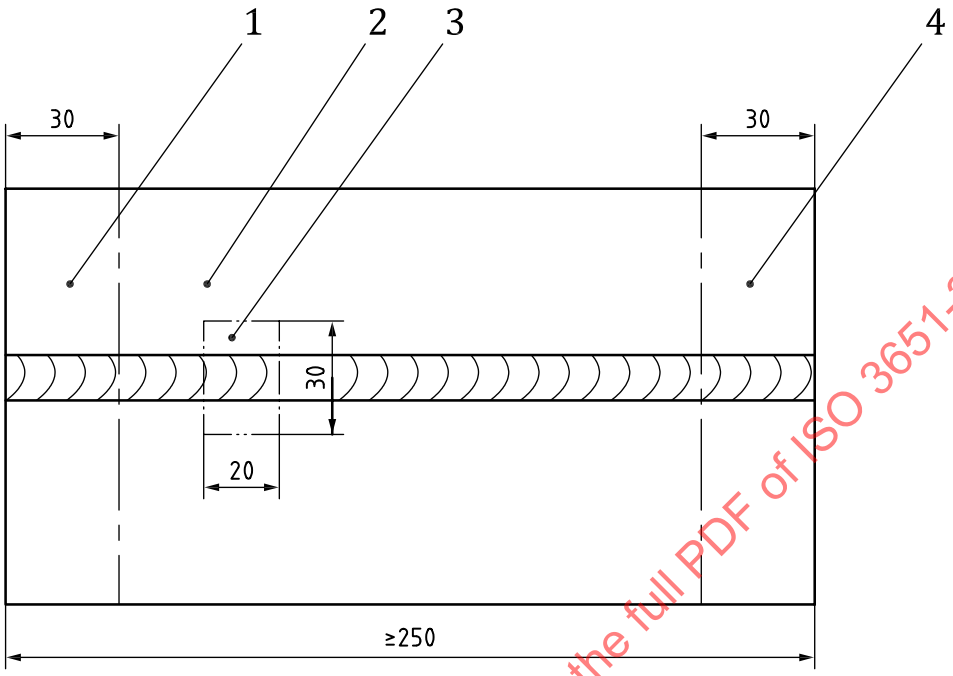
4.1 Principle

Depending on the purpose of the test and agreement between supplier and purchaser, the test pieces shall be prepared as specified in 4.2, 4.3 or 4.4. The thickness of the test specimen can be the same as the original samples.

4.2 Dimensions

A specimen having a total surface area of 5 cm^2 to 20 cm^2 is recommended. It should include welded zone, heat-affected zone and unaffected base metal.

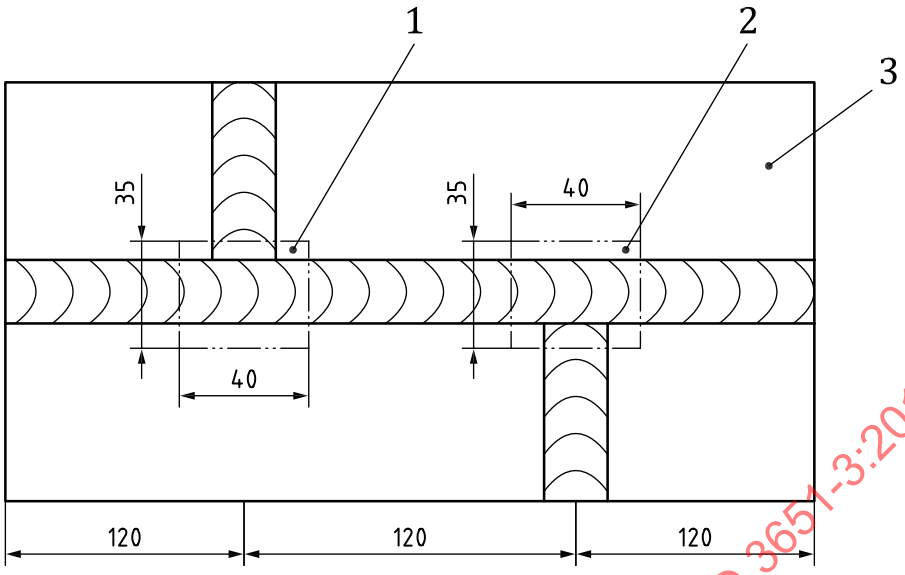
Dimensions in millimetres



Key

1 no sampling area (avoid weld defects)
2 sampling area
3 welding sample
4 no sampling area

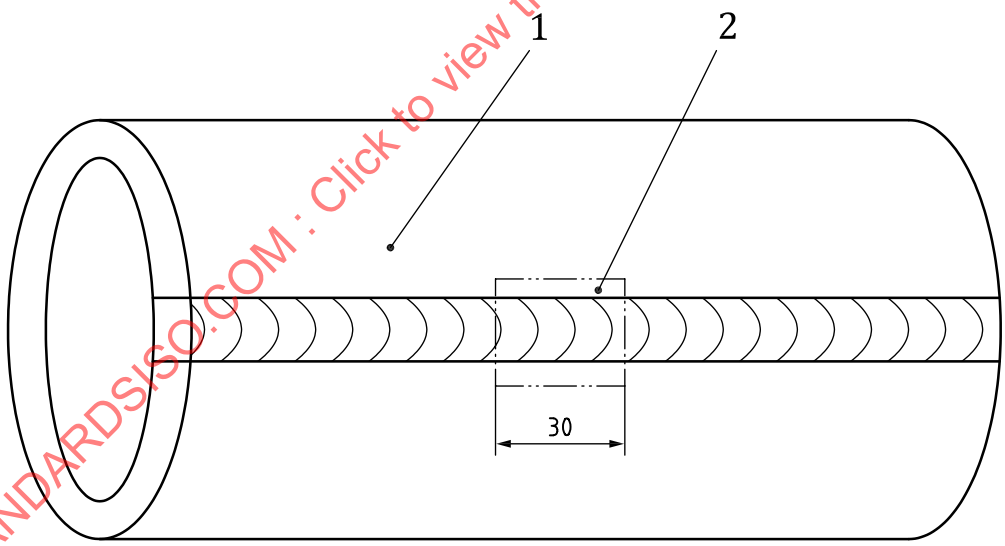
Figure 1 — Test piece for plates and strips with welded butt joint



Key

1 welding sample
2 welding sample
3 sampling area

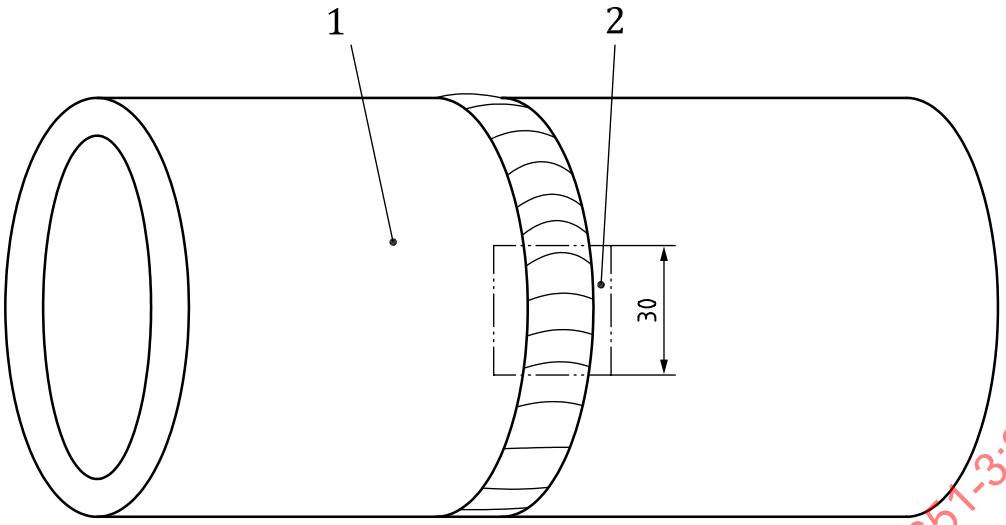
Figure 2 — Test piece for plates and strips with crossing welded butt joint



Key

1 sampling area
2 welding samples

Figure 3 — Longitudinal test piece for tubes with welded butt joint



Key

1 sampling area

2 welding samples

Figure 4 — Circumferential test piece for tubes with welded butt joint

4.3 Welded test pieces

Welded test pieces are prepared according to Figures 1 to 4.

As-received heat treatment shall be specified.

4.4 Sensitization heat treatment

In order to verify the intrinsic resistance to intergranular corrosion, it is necessary to carry out a sensitization heat treatment for low-Cr ferritic stainless steel as follows:

Step1: heating the specimen at 1 200 °C ± 10 °C for 30 min followed by water cooling;

Step2: heating the specimen at 600 °C ± 10 °C for 30 min followed by water cooling.

4.5 Mechanical preparation

The test pieces shall be descaled mechanically by grinding in the longitudinal plane on all surfaces and sharp edges with grade 600 or finer, iron-free abrasive paper or cloth.

Over-heating of the test pieces shall be avoided.

4.6 Degreasing and drying

The test piece shall be degreased using a suitable degreasing agent such as acetone or ethanol and dried before being placed into the corrosion solution.

5 Test methods

5.1 Apparatus

5.1.1 A flask, with a capacity of 1 l with a condenser.

5.1.2 **Heating device**, to keep the solution boiling.

5.1.3 **Metallographic microscope,** magnification of ~×500.

5.2 Corrosive solution

5.2.1 The test solution shall be prepared as follows.

5.2.2 Weigh 363 g of analytical reagent copper sulfate ($CuSO_4 \cdot 5H_2O$) and add into the flask. The test pieces are covered in electrolytic grade copper filings on the bottom of the flask. The amount of copper shall be at least 50 $g \cdot l^{-1}$ of the solution.

5.2.3 Measure 600 ml of distilled water and pour into the flask.

5.2.4 Measure 5,0 g of the reagent grade sulfuric acid of a density at an ambient temperature of 1,84 $g \cdot ml^{-1}$. Add the acid slowly to the solution in the flask to avoid boiling by the heat evolved.

5.2.5 Fit the condenser to the flask and circulate cooling water.

5.2.6 Boil the solution until all the copper sulfate is dissolved.

5.2.7 Based on the above steps, the solution of 0,5 % H_2SO_4 + 24 % $CuSO_4$ is now prepared.

5.2.8 The corrosive solution shall be used only once.

5.3 Copper addition

5.3.1 A sufficient quantity of electrolytic grade copper with high surface area (e.g. shavings or filings) shall be used to cover all surfaces of the specimens which are embedded in the copper on the bottom of the flask.

5.3.2 The amount of copper shall be at least 50 g/l of solution to assure galvanic coupling between copper and test specimens.

5.3.3 The copper may be reused if it is cleaned in warm deionized water after each test.

5.4 Procedure

5.4.1 The volume of acidified copper sulfate solution should be sufficient to completely immerse the specimens and provide a minimum 8 ml/cm^2 of the total surface area of the test specimens.

5.4.2 As many as three specimens being of the same grade can be tested in one flask. The different grade steels or the same grade with different processing history should not be tested in the same flask.

5.4.3 The test specimens shall be immersed in the test solution at ambient temperature which is then brought to a boil and maintained boiling during the test period.

5.4.4 Begin the test period when the test solution reaches the boiling point. The solution shall be boiled for 15 h ± 3 h. The test duration shall be recorded.

5.4.5 The test specimen is rinsed in deionized water and then dried.

6 Evaluation by metallographic examination

6.1 Examine original surface of the test specimen under a microscope at ~×500 magnification. Grain dropping or grain boundaries corroded completely are usually indication of intergranular attack.

6.2 Cut the sample in cross-section and prepare the metallographic sample.

6.3 Examine the cross-section under a microscope. The depth of intergranular cracks that can be tolerated is subject to agreement between the supplier and purchaser. The material may be reported as being resistant to intergranular corrosion if the maximum depth of corrosion shall be not more than 5 µm.

7 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 3651-3;

b) steel grade including chemical composition;

c) method of preparation;

d) nature of the test piece;

e) nature of the as-delivered heat treatment and the sensitization heat treatment, when applied;

f) results of the test;

g) any incident which may have an effect on the results.