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Austenitic stainless steels — Determination of resistance to intergranular corrosion — Part II : Corrosion test in a sulphuric acid/copper sulphate medium in the presence of copper turnings (Monypenny Strauss test)

Aciers inoxydables austénitiques — Détermination de la résistance à la corrosion intergranulaire — Partie II : Essai de corrosion en milieu acide sulfurique/sulfate de cuivre en présence de copeaux de cuivre (Essai dit de Monypenny Strauss)

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3651/II was drawn up by Technical Committee ISO/TC 17, *Steel*, and was circulated to the Member Bodies in February 1975.

It has been approved by the Member Bodies of the following countries :

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The Member Body of the following country expressed disapproval of the document on technical grounds :

U.S.A.

Austenitic stainless steels — Determination of resistance to intergranular corrosion — Part II : Corrosion test in a sulphuric acid/copper sulphate medium in the presence of copper turnings (Monypenny Strauss test)

1 SCOPE

This International Standard specifies a method for the determination of the resistance to intergranular corrosion of austenitic stainless steels in a sulphuric acid/copper sulphate medium in the presence of copper turnings (Monypenny Strauss test). It also specifies the purposes which may be assigned to the test.

2 FIELD OF APPLICATION

The method is applicable only to austenitic stainless steels supplied in the form of cast, rolled or forged products and tubes and intended to be used in a mildly oxidizing acid medium (for example, sulphuric acid, phosphoric acid).

NOTE — 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 may not be used to check resistance to other forms of corrosion (general corrosion, by pitting, stress corrosion, etc.). It is necessary for the user to adapt the specified corrosion test to the use which will be made of the metal. This test should, in no case, be considered as an absolute criterion of the quality of the metal.

3 GENERAL

3.1 The term "intergranular corrosion test" denotes the corrosion test carried out by means of the preferential attacking of the grain boundaries.

Austenitic stainless steels may be subject to such an attack when they are kept at a temperature between about 500 and 800 °C. This heat cycle, which may provoke sensitization to intergranular corrosion, may occur during hot-forming (forging, rolling), as the result of incorrect solution treatment or during a welding operation.

NOTE — In the field of application of this test, the intergranular corrosion may be connected with the presence along the grain boundaries of chromium-depleted regions due, in general, to precipitation of chromium carbides.

3.2 The interpretation of the results shall form the subject of an agreement between the interested parties.

4 PURPOSE OF THE TEST

This intergranular corrosion test may have either of the purposes given in 4.1 and 4.2. If the order specifies this corrosion test, the purpose of the test shall be stated at the time of ordering.

4.1 Verification of the intrinsic resistance of the metal to intergranular corrosion

This verification applies only to steels defined in ISO 683/XIII[1], specified specially for resistance to intergranular corrosion (low carbon steels : C ⩽ 0,03 %, and stabilized steels).[2] The metal is inspected after having undergone a heat treatment for sensitization. (See clause 5.)

4.2 Inspection of the efficiency of the solution treatment

This inspection is only carried out on thin products for which the cooling speed may be made sufficiently rapid. It is only of interest for the steels which are not defined in 4.1. The metal is inspected in the state in which it is delivered to the user, without heat treatment for sensitization.

5 HEAT TREATMENT FOR SENSITIZATION

In order to verify the intrinsic resistance to intergranular corrosion (see 4.1), it is necessary to carry out a heat treatment for sensitization for stabilized steels and steels with a very low carbon content. This sensitization treatment is usually obtained by maintaining the test piece for 30 min at a temperature of 700 ± 10 °C followed by rapid cooling (in water).

Other sensitization treatments, for example for the preparation of welded test pieces, may be provided for by agreement between the interested parties.

1) ISO 683/XIII, *Heat-treated steels, alloy steels and free-cutting steels — Part 13 : Wrought stainless steels.*

2) By agreement between the interested parties, this test can equally be applied to steels having a maximum carbon content of 0,07 %.

6 CORROSION TEST

6.1 Principle

A test piece, prepared as specified in 6.4.2, is immersed in a boiling sulphuric acid/copper sulphate solution for a specified time (see 6.5). The test piece is then subjected to a bend test. The convex surface of the test piece is examined after bending in order to reveal any cracks.

When a test is made on a seamless tube, the test piece being subjected to the corrosion test is cut to give a test piece suitable for bending, and the surface of the tube to be put in tension during the bend test is to be specified on the order.

In the case of certain tubes (for example small diameter tubes), a flattening test is used instead of the bend test.

6.2 Corrosive solution

The sulphuric acid/copper sulphate solution shall be prepared as follows, using analytical quality reagents.

Dissolve 100 g of copper(II) sulphate pentahydrate ($CuSO_4.5H_2O$) in 700 ml of distilled water. Then add 184 g (100 ml) of sulphuric acid (ρ_{20} 1,84 g/ml) and make up to 1 000 ml with distilled water.

The corrosive solution may only be used once.

6.3 Apparatus

6.3.1 Conical flask, capacity 1 l, fitted with a 4-ball rising condenser.

6.3.2 Support for the test piece, generally of glass, if necessary.

6.3.3 Heating device, to keep the solution boiling.

6.4 Test piece

6.4.1 *Dimensions*

The test piece shall have a total surface area of 15 to 35 cm^2 and may vary according to the type of product. In the case of sheets with a thickness greater than 4 mm, the test piece shall have a maximum thickness of 4 mm and one of the rolled surfaces shall be retained.

In the case of tubes up to 40 mm outside diameter, the test piece shall be a full cross-section of the tube and will be subjected to a flattening test (see 6.6).

For cast products, the test pieces are selected from test blocks as defined in ISO . . ., *Steel castings — General technical delivery conditions.*[1] In case of dispute, a flat test piece shall be used having the following dimensions :

- thickness between 2 and 5 mm;
- width at least 20 mm;
- length at least 50 mm.

Test pieces for welded products shall be the subject of agreement between the interested parties.

6.4.2 *Preparation*

Depending on the purpose of the test (see clause 4), the test piece, either with or without sensitization treatment, shall be prepared as specified in either 6.4.2.1 or 6.4.2.2. Unless otherwise stated on the order, the method of preparation shall be left to the manufacturer.

6.4.2.1 MECHANICAL PREPARATION

The test piece shall be descaled mechanically by polishing on all surfaces with grade 120 abrasive paper or cloth.

6.4.2.2 CHEMICAL PREPARATION

The test piece shall be descaled, without any previous mechanical treatment, in a solution of 50 volumes of hydrochloric acid (ρ_{20} 1,19 g/ml), 5 volumes of nitric acid (ρ_{20} 1,40 g/ml) and 50 volumes of water at 50 to 60 °C.

6.4.2.3 DEGREASING

The test piece shall then be degreased before being placed in the corrosive solution.

6.5 Procedure

6.5.1 *Immersion in corrosive solution*

Carry out the test in the presence of metallic copper (50 g of copper filings per litre of solution, introduced at the beginning of the test), using a volume of corrosive solution of at least 10 ml per square centimetre of surface area of the test piece. Place the test piece on the copper filings, bring to the boil and continue boiling for 15 to 24 h. In cases of dispute the duration of the test shall be the subject of an agreement between the interested parties. For cast products, the duration of the test, in case of dispute, shall be 20 h.

Then remove the test piece and wash it in water.

Several test pieces may be tested at the same time in the same receptacle, provided that there is no contact between them.

1) In preparation.

6.5.2 *Bend test*

In the case of cylindrical and flat test pieces, subject the test piece to a bend test at 90° on a mandrel, the radius of which is equal to the thickness of the test piece, except for cast products in which case the mandrel radius is twice the thickness of the test piece.

6.6 Evaluation

The surface of the test piece (convex side) shall be examined with the naked eye or using a magnifying glass (magnification not greater than x 10), in order to detect cracking.

In the case of sheet with a thickness greater than 4 mm, the convex surface of the test piece shall be the retained rolled surface.

In the case of tubes up to 40 mm outside diameter, the distance between the platens after flattening, measured under load, shall be not greater than the following value, H, in millimetres :

$$H = \frac{1{,}09\,Dt}{0{,}09\,D + t}$$

where

t is the specified wall thickness, in millimetres;

D is the outside diameter of the tube, in millimetres.

In the case of welded tube, the weld shall be at the point of maximum flattening.

NOTE – In the case of doubtful results, a 90° bend test shall be carried out on a second test piece prepared in a similar way but without having been submitted to the corrosion test. The comparison of the two test pieces enables it to be established whether the cracks observed on the corroded test piece are due to intergranular corrosion.

At the same time, if cracks are observed which cannot be definitely identified as intergranular, the test pieces may be examined metallographically.