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Corrosion of metals and alloys —Corrosion test for intergranular corrosion susceptibility of low-Cr ferritic stainless steels

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Contents

Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75% of the member bodies casting a vote.

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Introduction

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

The low-Cr ferritic stainless steels may be subject to such attack when they are at a temperature between about *500* ℃ to *1300* ℃. This heat cycle, which may provoke sensitization to intergranular corrosion, may occur during forging, rolling or welding operation.

Low-Cr ferritic stainless steels may show high uniform corrosion rates and copper deposit when tested by the methods given by ISO *3651* because of electrochemical potential difference between the matrix and the Cr depletion is less than that of high-Cr ferritic stainless steels. This possibility must be considered in selecting this test method. Application of this standard test to the other stainless steels will be specific agreement between producer and user.

1 Scope

1.1 This International Standard specifies the determination of the intergranular corrosion susceptibility of low-Cr (less than *16 %* Cr) ferritic stainless steels in the *0.5 %* sulfuric acid /copper sulfate test. It also specifies the purposes which may be assigned to the test.

1.2 The methods are applicable to stainless steels supplied in the form of cast, rolled or forged products and tubes and intended for use in a mild oxidizing acid medium.

2 Normative references

The following referenced documents are indispensable for the application 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 *3651* (all parts), Determination of resistance to intergranular corrosion of stainless steels

ISO *21610*, Corrosion of metals and alloys — Accelerated corrosion test for intergranular corrosion susceptibility of austenitic stainless steels

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.

4 Test specimen

4.1 Dimensions

The test piece shall have a total surface area of *15 cm²* to *35 cm²*. In the case of sheets with a thickness greater than 6 mm, the test piece shall have a maximum thickness of *6 mm* and one of the rolled surfaces shall be retained.

The test pieces are selected from the product as defined in the product standard. In case of dispute, a flat test piece, when possible, shall be used having the following dimensions. These dimensions shall be used in relation to the available bending apparatus:

— thickness between *2 mm* and *6 mm*;

— width at least *10 mm*;

— length at least *50 mm*.

4.2 Welded test pieces

Welded test pieces are prepared as follows. For flat products, two pieces about *100* mm long and about *50 mm* wide are welded together and the test piece is cut according to figure *1*.

For tubes with a circumferential weld, the test piece is cut according to figure *2*.

When four pieces are welded together to form a cross weld, the first-laid weld bead shall be in the longitudinal direction of the test piece as shown in figure *3*.
The retained surface shall form the convex side of the test piece after bending.
For welded tubes with outer diameter over *60 mm*, the test pieces with length of at least *50 mm* and width of at least *20 mm* are prepared as shown in figure *4*.
For welded tubes up to *60 mm* outside diameter, the test piece shall be a full cross section of the tube and will be subject to a flattening test .

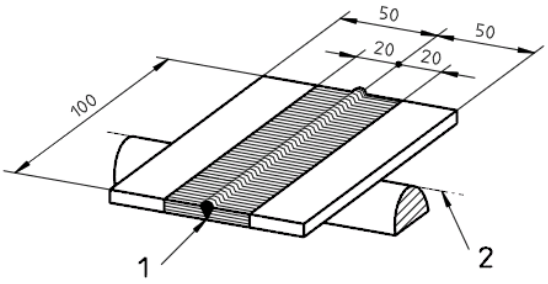

Figure *1* — Test piece for testing of plates and strips with welded butt joint（*1* Weld *2* Axis of the bending mandrel）

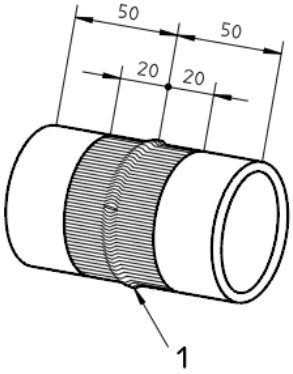

Figure 2 — Test piece for testing of tube with welded butt joint（*1* Weld）

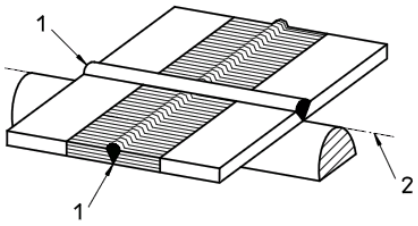

Figure *3* — Test piece for testing of plates and strip with cross welded butt joint（*1* Weld *2* Axis of the bending mandrel）

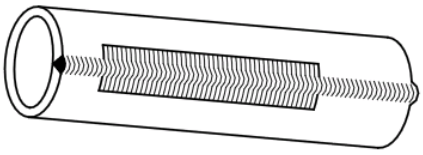

Figure *4* — Test piece for testing the longitudinal weld of welded tubes

4.3 Sensitization treatment

4.3.1 In order to verify the intrinsic resistance to intergranular corrosion, it is necessary to carry out a heat treatment for sensitization for stabilized steels and steels with low carbon content. If to be used as-delivered, shall be tested using specimens made from metal taken from the production line and the sensitization heat treatment may be carried out with the agreement of the customer. Either the samples from which the specimens are made or the specimens themselves may be given the

sensitization heat treatment.

If discrepancies arise in the interpretation of the test results, the samples shall be subjected to a sensitization heat treatment procedure. To do so, degreased samples shall be placed in a furnace which has been pre-heated to the sensitization temperature.

The sensitization heat treatment can be: Heating the specimen at *1200 ℃± 10 ℃* for 30min followed by water cooling; then heating the specimen at *600 ℃ ± 10 ℃* for 30min followed by water cooling.

4.3.2 Steels which are to be used in a cold-worked state or in a semi-cold-worked state shall be tested using specimens that have not been given a sensitization heat treatment.

4.3.3 At companies which manufacture welded joints, test specimens from weldments can be used without sensitization heat treatment if the welding conditions have been properly controlled.

4.3.4 Where Stainless steels are to be tested after repeated heat treatment which differs from the sensitization heat treatment, testing shall be carried out in accordance with this subclause (4.3) as for the testing of a new batch of metal.

4.3.5 In cases when, during the manufacture of an item, control-welded joints are subjected to heat treatments that can influence their properties, the test specimens shall be treated under similar conditions.

4.3.6 Scale that forms on the test specimen surface during hardening or sensitization heat treatment shall be removed by chemical or electrochemical etching or by a mechanical process before grinding and polishing. The depth of any surface treatment shall not exceed 1 mm.

Chemical etching shall be carried out either in the following solution:

— *620 ml* ± *3 ml* of HNO3 (ρ20 = *1,35 g/ml*);

— *76,0 g* ± *0,1 g* of NH4F;

— *300 ml* ± *3 ml of* water*;*

— temperature *20 °C* ± *5 °C*;

or electrolytically as follows:

— *34 ml* ± *1 ml* of orthophosphoric acid (ρ20 = *1,68 g/ml*);

— *11 ml* ± *1* ml of HNO3 (ρ20 = *1,35 g/ml*);

— *955 ml* ± *3 ml* of water;

— temperature *40 °C* to *50 °C*;

— current density *0,5* × 10^4 $A \cdot m^2$ to *0,6* ×10^4 $A \cdot m^2$.

The test specimen shall be etched or ground to completely remove any scale. After etching, the specimens shall be carefully rinsed with water. Other etching solutions and etching regimes may be used for scale removal if they provide complete scale removal and, for steels that are resistant to intergranular corrosion, also prevent both preferential etching along the grain boundaries and pitting corrosion. Where discrepancies arise in the test results, etching shall be carried out only with the

solutions prescribed above.

If the test specimen remove the scale by griding mechanically in the longitudinal on all surface and sharp edge with iron-free abrasion paper or cloth.

4.4 The roughness of the controlled surfaces of test specimens prior to testing shall not exceed *0.8 µm*. The required finish shall be achieved by polishing or grinding. Overheating of the test piece shall be avoided.

Test specimens which are produced from tubes that have been subjected to cold or hot deformation or from cold rolled or cold-drawn metal products or from metal products with special surface treatments shall not be subjected to polishing or grinding unless this is called for in the technical documentation.

4.5 Specimens shall be marked before testing. The identification marks shall be applied either by stamping or by electrical discharge machining of brittle material at one or both ends of the specimen at a distance of *5 mm* to *10 mm* from the edge.

4.6 Before testing, the specimens shall be degreased using suitable reagent grade organic solvents. If the specimens are placed in the test vessel directly after the completion of etching or rinsing procedure, the degreasing procedure can be omitted.

5 Test methods

5.1 Apparatus

Erlenmeyer flask, capacity *1 L*, or other suitable flask of optional capacity fitted, with an Allihn condenser with at least four balls. Heating device, to keep the solution boiling.

5.2 Corrosive solution

The test solution shall be prepared as follows using analytical quality reagents.

Dissolve *200 g* of copper (II) sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) in *700 ml* of distilled water. Then add *5 g* (*2,8 ml*) of sulfuric acid (ρ_{20} = *1,84 g/ml*) and make up to *1000* ml with distilled water.

5.3 Procedure

More than one test piece may be tested in each flask provided that the volume of solution is at least 8 $ml \cdot cm^{-2}$ of the total surface area of the test pieces. The test pieces are embedded in electrical grade copper filings on the bottom of the flask. The amount of copper shall be at least *50 g/l* of solution. The test pieces shall be in metallic contact with the copper but not with each other. The test pieces are firstly immersed in the cold test solution following which the solution is brought to the boil, from which point on wards the test duration commences. The solution shall be maintained at boiling *16 h ± 2 h*. In case of dispute, the duration of the test shall be *16 h*. The copper filings may be reused if they are cleaned in warm tap water after each test. The corrosive solution shall be used only once.

6 Evaluation of intergranular corrosion

6.1 Bend test

Cylindrical and flat test pieces from wrought products shall be subject to a bend test after corrosion test. The test piece shall be bent to at least *90°* over a mandrel with a radius not exceeding the thickness of the test piece. For cast products, the radius of the mandrel shall be no more than twice the thickness of the test piece.

Tubes with up to *60 mm* outside diameter shall be subject to a flattening test. The distance between the platens after flattening, measured under load, shall be no greater than the following value, H, in millimetres:

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

Where

T is the specified wall thickness, in millimetres;

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

Welded tubes shall have the longitudinal weld at the point of maximum strain except when the purpose of the test is to test a cross weld according to figure *2*.

NOTE — A determination of the weight loss may be used as a complement to the bend test or the flattening test for tubes with up to *60 mm* outside diameter. It is to be noted, however, that the weight loss will not measure the intergranular corrosion only but also any general corrosion occuring.

6.2 Evaluation

6.2.1 The bent specimens shall be examined using a magnifying glass with a magnification of *5* to *20* times.

6.2.2 When cracks are not found in specimens which have been bent after testing ,apart from longitudinal cracks and cracks directly on the edges, the material is considered to be resistant to intergranular corrosion. However, if cracks are found, testing may be repeated, this time in accordance with ISO *21610-2009*.

6.2.3 In cases when, after exposure in the test solution and during or after bending, the control specimens break or cracks are found in specimens which were bent after testing,apart from longitudinal cracks and cracks directly on the edges, or the specimen thickness is less than *0,1 mm* and intergranular corrosion is not found or it is impossible to bend the specimen because of its size, then the intergranular corrosion susceptibility shall be evaluated by metallography.

6.2.4 Before metallographic evaluation of the intergranular corrosion susceptibility of specimens that have undergone corrosion testing, the specimen for metallographic examination shall be cut out of the corrosion tested specimen along a cutting plane perpendicular to the specimen surface under assessment. In the case of metallographic specimen preparation from welded specimens, the cutting line shall be perpendicular to the weld line and the cutting plane shall include both the weld metal and the metal of the heat-affected zone.

The recommended metallographic specimen length from the surface to be assessed shall be *15 mm* to *20 mm*.

The cut surface shall be coincident with the metallographic specimen surface.
The metallographic specimen shall be produced in such a way that no corner breaks or fins are generated.
6.2.5 To reveal intergranular corrosion and evaluate the depth of any corrosion-related cracking, previously etched metallographic specimens shall be examined at a magnification of *200* times. The etching shall produce only slight attack of the grain boundaries.
The surfaces under investigation of the metallographic specimens shall be examined. The maximum depth of attack shall be evaluated for six different fields of view. The sites with the greatest depths of intergranular corrosion shall be included in these fields of view.
6.2.6 The material may be reported as being resistant to intergranular corrosion if the metallographic assessment reveals the maximum depth of corrosion attack to be not more than 10 µm, unless the specifications for the metal.
products contain other requirements. For specimens produced from metal products with thicknesses of less than *1,5 mm*, the maximum depth of corrosion shall be not more than *5 µm*.

7 Test report

The following information shall be included in the test report:
a) the steel grade;
b) the type of metal product;
c) the method of manufacture of the product;
d) the heat treatment used;
e) the test specimen type and size;
f) the test method used;
g) the result of the test, i.e. whether the specimens are resistant to intergranular corrosion or not resistant to intergranular corrosion.

Annex A Comparison of ISO *3651-2:1998* and this new standard

	ISO *3651-2*	this new standard
Application	Steels with more than *16 %* Cr	Steels with less than *16 %* Cr
Solution A	*100 g* $CuSO_4.5H_2O$ + *184 g* H_2SO_4	*200 g* $CuSO_4.5H_2O$+ *5* g H_2SO_4
Solution B	*110 g* $CuSO_4.5H_2O$+*460 g* H_2SO_4	/
Solution C	/	/
Exposure duration	*20 h* ± *5 h*	*16h* ± *2 h*
Sensitizing treatment	/	*1200* ℃, for *30 min,* then *600* ℃,for *30 min*
Angle of bending	more than *90°*	more than *90°*
Bending mandrel diameter	t	t
Bending Examination magnification	Approx. *10*	*5-20* ×
Metallographic,Examination magnification		*200* ×