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Corrosion of metals and alloys -- Accelerated corrosion test for intergranular corrosion susceptibility of austenitic stainless steels

金属和合金的腐蚀 奥氏体不锈钢晶间腐蚀敏感性加速腐蚀试验方法

(ISO 21610:2009, MOD)

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Warning -- Some of the reagents used in this test method are corrosive and shall be handled with care. If the skin is splashed by it, rinse immediately with water; severe cases shall be treated immediately.

1 Scope

This document specifies an accelerated test method for determining the susceptibility to intergranular corrosion of austenitic stainless steel.

This document applies to the testing of a variety of metal products, including double rolled metal, welded joints, deposited metal, and weld metal.

Note 1: The results obtained by this method are the same as those obtained by the methods in the ISO 3651-1 and ISO 3651-2 standards, but the accelerated corrosion test cycle is shorter.

Note 2: This document specifies two test methods:

--- Method A: Corrosion test in copper sulfate and concentrated sulfuric acid solutions in the presence of metallic copper;

--- Method B: Corrosion test in copper sulfate, sulfuric acid, and copper fluoride solutions in the presence of metallic copper.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) is applicable to this standard.

GB/T 10123 Corrosion of metals and alloys -- Basic terms and definitions (GB/T 10123-2001, ISO 8044:1999, IDT)

ISO 3651(all parts) Determination of resistance to intergranular corrosion of stainless steels

a) For plate specimens, segmental specimens, cylindrical specimens with a diameter not exceeding 5 mm, and tubular specimens with a diameter not exceeding 5 mm, angle bending should be (90±3)°;

b) Annular and tubular specimens with a diameter exceeding 5 mm should be flattened and formed into cones;

c) Examine the curved surfaces in cross-sectional regions with sufficient thickness to identify intergranular cracks.

4.4 Machining the blank to prepare the specimen of the required thickness:

a) The metal plate should be sampled from one side only; if one of the sides is treated, then take samples from the treated side;

b) The rolled metal (rolled sections and profiles), forgings, castings, and tubular samples should be sampled from a suitable surface;

c) The hot-deformed or cold-deformed pipe should be sampled from the outside;

d) The hot-rolled pipe should be sampled from the inner or outer surface;

e) The pipe that has been exposed to the service environment should be sampled from the side that is in contact with the service environment.

4.5 The following kinds and types of specimens should be prepared from welded joints (see Table 2):

--- Prepare Type I or Type II plate specimens from welded joints of plate metal, rolled sections, castings or forgings;

--- Prepare Type II annular, tubular, or segmental specimens from electric welded pipes;

--- Prepare Type I segmental, annular, or tubular specimens from girth welded joints of pipe sections;

--- Prepare Type I or II plate specimens, or Type I segmental, annular, or tubular specimens from welded joints of double-wall tubes.

4.6 For welded specimens (Table 2, Type I and Type II), the weld reinforcement should be removed with a depth not exceeding 1 mm.

The required thickness of the specimen should be achieved by machining, and the metal should be removed from the side that is not in contact with the corrosive medium. If it is not sure which side is in contact with the corrosive medium, the machining should be processed on the side where the weld metal is least heated during the welding process.

If it is necessary to reduce the wall thickness of the specimen, sampling should be carried out in accordance with 4.4.

4.7 The inspection of electrodes, filler wires, and strips shall be carried out through the fabricated surfacing or weld metal.

The surfacing metal shall be tested with plate specimens (see Table 2). The specimen shall be cut from the multi-layer surfacing metal made of the inspected welding material (the surfacing metal substrate is not used for specimen preparation, and can be prepared from materials with similar chemical compositions). When the chemical composition of the metal substrate is similar to that of the surfacing metal, the number of surfacing metal substrates not used for specimen preparation can be reduced to 3 layers.

The weld metal is tested with plate specimens cut from the upper layer of the multi-layer welded seam of the welded joint. The welded seam thickness of the cut specimen area should not be less than 15 mm. The chemical composition of the plate used for the welded joint should be similar to the chemical composition of the weld metal.

Welded backing plates can be used when using thin plates to obtain the welded seam of desired thickness. The chemical composition of the steel for welded backing plate is similar to the thin plate. When welding the edge of the thin plate specimen initially, the entire surface of the welded backing plate should be similarly treated.

Weld metal testing should be carried out on plate welded specimens (see Types I and II in Table 2) prepared from specially-made welded joints. The welded joints are made of intergranular-corrosion-resistant steel and welding consumables to be evaluated.

4.8 The recommended size of the specimen is shown in Appendix B, and different specimen sizes can also be used.

4.9 When testing plates, pipes, metal profiles, wires, and strips, at least two specimens should be prepared for each group.

For all types of steels and alloys, at least 4 specimens (two of which are used for comparison) should be prepared for each forging, casting, and weldment.

The control specimen should be bent 90°±3° and not be exposed to the boiling test solution. If the specimen needs to be inspected from both sides, then the specimen should be bent into a “Z” shape.

4.10 The sensitization treatment of unstabilized steel is as follows: on the premise of no special requirements in the technical documents, if the unstabilized steel does not contain titanium or niobium, the maximum carbon content is greater than 0.03% and it is used under the delivery conditions, then the sensitization treatment test should be carried out on the specimen of the delivery conditions.

- (11±1)mL HNO_3 (ρ_{20}=1.35 g/mL);
- (955±3)mL H_2O;
- Temperature: 40 °C~50 °C;
- Current density 0.5×10^4 A • m^{-2}~0.6×10^4 A • m^{-2}.

The oxide coating on the specimen should be thoroughly removed, and the specimen should be carefully rinsed with water after etching.

It can be removed by other etching solutions or methods that can ensure the complete removal of the oxide coating. For intergranular corrosion-resistant steels, it is necessary to prevent preferential erosion or pitting along the grain boundaries.

When there are any differences in the test results, only the above solutions should be used for etching.

4.12 During the polishing or grinding process of the specimen, the surface should be prevented from overheating, and the surface roughness of the processed specimen should not exceed 0.8 μm.

Unless otherwise stated in the technical documents, specimens made of cold-deformed and heat-deformed tube or cold-drawn metal products, or made of metal products with special surface treatment, should not be polished or ground.

4.13 Mark the specimen before testing. At one or both ends of the specimen, the position with a distance of 5 mm~10 mm from the edge shall be marked by means of steel stamping or electrical discharge machining.

4.14 Degrease the specimen with a suitable organic solvent (reagent grade) before testing. The degreasing step may be omitted if the specimen is placed directly in the test vessel after the etching or rinsing step.

5 Test methods

5.1 Method A

5.1.1 Method principle

The steel specimen shall be placed together with the metallic copper in a solution made up of H_2SO_4 and $CuSO_4 \cdot 5H_2O$, and the concentration of which shall be higher than the concentration of the solution extracted by the method specified in ISO 3651-2.

This method is an accelerated test method and can be used for the verification of steel grading specified in ISO 3651-2.

5.1.2 Test solution

Use analytical reagents to prepare the test solution: add (50±0.1) g of $CuSO_4 \cdot 5H_2O$ to (1000±3) mL of water, and then gradually add (250±3) mL of H_2SO_4 (ρ_{20}=1.84 g/mL).

5.1.3 Test steps

5.1.3.1 The test shall be carried out in a glass flask with a condenser tube, or a container made of corrosion-resistant stabilized chromium-nickel steel with a lid and a condenser tube. Spread a layer of copper chips on the bottom of the reaction vessel, and then place the specimen in.

The following operations can be performed:

a) Use a perforated copper plate with a sharp edge (bore diameter shall be 5 mm~7 mm) to replace the copper chips, so that the upper and lower sides of the specimen can be fully contacted with the copper plate; it helps to ensure that the test solution can fully rinse the sample and the corrosion products are well removed. If there is a discrepancy in the assessment of metal quality, copper chips should be used for the test.

b) When the two sides of the specimen are in full contact with the copper chips or copper plate, a multi-layer of specimens can be placed. The test solution should be at least 20 cm higher than the uppermost specimens, copper chips, or copper plate.

After the solution is poured into the container, heat it to the boiling point and boil it, see 5.1.3.3 for details.

5.1.3.2 The heating of the condenser tube shall be avoided.

5.1.3.3 The boiling time shall be (8.00±0.25) h. In the case of forced interruption of the test, the specimen should not be taken out of the test solution. The test time is the sum of all boiling periods.

5.1.3.4 If there is a difference in the results of the interrupted test, it is advisable to use a glass flask and continuously expose the specimen to the boiling solution to repeat the test. At this time, the amount of solution calculated according to the surface area of the specimen should not be less than 10 mL/cm^2.

5.1.3.5 After the test is finished in the boiling solution, rinse the specimen with tap water and blow-dry it. Use 20%~30% nitric acid solution to remove the copper attached to the surface of the specimen that cannot be washed away with water.

5.1.3.6 The test solution without discoloration can be reused. The exception is when there is a dispute between the parties involved over the experimental results.

6.8 Check the bent specimen with a magnifying glass of 8 times to 12 times.

6.9 If no cracks (except longitudinal cracks or edge cracks) are found in the bent specimen after the test, the material can be considered to be resistant to intergranular corrosion. If cracks are found, the test can be repeated according to ISO 3651-2.

6.10 After exposure to the test solution and during or after bending:

a) The control sample is broken;

b) Cracks are found on the bent specimen after the test (except for longitudinal cracks or edge cracks);

c) The thickness of the specimen is less than 0.1 mm, and no intergranular corrosion is found;

d) Due to the size, the specimen cannot be bent.

Intergranular corrosion susceptibility shall be assessed by metallographic methods.

6.11 Before performing the intergranular corrosion susceptibility evaluation on the specimen after the corrosion test, the specimen for the metallographic examination should be cut from the cutting plane that is perpendicular to the tested surface of the corrosion specimen. For the metallographic specimen prepared from the welded specimen, the cutting line should be perpendicular to the welding fusion line, and the cutting section should include the weld metal and the heat-affected zone metal. The recommended length of the metallographic specimen (calculated by the surface to be measured) is 15 mm~20 mm. The cutting section should meet the surface requirements of the metallographic specimen. Metallographic specimens should be prepared in a manner that does not produce corner breakage or burrs.

6.12 In order to show intergranular corrosion and evaluate the depth of corrosion cracks, the metallographic specimens are inspected under a magnification of 200 after etching. Erosion should only slightly corrode the grain boundaries. See B.2 for recommended etching solutions and procedures. The entire surface area of the metallographic specimen to be tested shall be examined. The maximum depth of corrosion shall be assessed from 6 different fields of view. These fields of view shall contain the location of the maximum intergranular corrosion depth.

6.13 If the maximum corrosion depth obtained by metallographic evaluation does not exceed 30 μm, the material is resistant to intergranular corrosion (unless otherwise required by the metal product specification). For metal product specimens with a thickness of less than 1.5 mm, the maximum depth of corrosion should not exceed 10 μm.

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