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Corrosion of metals and alloys - Test methods for intergranular corrosion of austenitic and ferriticaustenitic (duplex) stainless steels

金属和合金的腐蚀 奥氏体及铁素体

奥氏体 (双相) 不锈钢晶间腐蚀试验方法

[ISO 3651-1:1998, Determination of resistance to intergranular corrosion of stainless steels - Part 1: Austenitic and ferritic-austenitic (duplex) stainless steels - Corrosion test in nitric acid medium by measurement of loss in mass (Huey test); ISO 3651-2:1998, Determination of resistance to intergranular corrosion of stainless steels - Part 2: Ferritic, austenitic and ferritic-austenitic (duplex) stainless steels - Corrosion test in media containing sulfuric acid, MOD]

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Corrosion of metals and alloys - Test methods for intergranular corrosion of austenitic and ferriticaustenitic (duplex) stainless steels

1 Scope

This Standard specifies the samples, test solutions, test instruments and equipment, test conditions and procedures, test result evaluation and test report of test methods for intergranular corrosion of austenitic and ferritic-austenitic (duplex) stainless steels.

This Standard applies to the inspection of intergranular corrosion tendency of austenitic stainless steels and ferritic-austenitic (duplex) stainless steels (hereinafter referred to as duplex stainless steels). It includes the following test methods:

a) Method A: 10% oxalic acid corrosion test method

It is a screening test to check the intergranular corrosion of austenitic stainless steels. After the sample is electrolytically etched in 10% oxalic acid solution, observe the metallographic structure of the etched surface under a microscope, so as to determine whether Method B, Method C, Method E and other long-time hot acid tests are required. In the case of not allowing damage to the tested structural parts and equipment, it can also be used as an independent intergranular corrosion inspection method.

b) Method B: 50% sulfuric acid-ferric sulfate corrosion test method

Place the austenitic stainless steels in the 50% sulfuric acid-ferric sulfate solution for a boiling test; then, use the corrosion rate to evaluate the intergranular corrosion tendency.

c) Method C: 65% nitric acid corrosion test method

Place the austenitic stainless steels in the 65% nitric acid solution for a boiling test; then, use the corrosion rate to evaluate the intergranular corrosion tendency.

d) Method E: copper-copper sulfate-16% sulfuric acid corrosion test method

Place the austenitic stainless steels and duplex stainless steels in the copper-copper sulfate-16% sulfuric acid solution for a boiling test; then,

use the bending method or the metallographic method to determine the intergranular corrosion tendency.

e) Method F: copper-copper sulfate-35% sulfuric acid corrosion test method

Place the austenitic stainless steels and duplex stainless steels in the copper-copper sulfate-35% sulfuric acid solution for a boiling test; then, use the bending method or the metallographic method to determine the intergranular corrosion tendency.

f) Method G: 40% sulfuric acid-ferric sulfate corrosion test method

Place the austenitic stainless steels and duplex stainless steels in the 40% sulfuric acid-ferric sulfate solution for a boiling test; then, use the bending method or the metallographic method to determine the intergranular corrosion tendency.

Appendix C of this Standard gives the characteristics of each test method in the form of a table; Appendix D gives the application examples of method E, method F, and method G.

The test methods in this Standard are not applicable to predicting the intergranular corrosion resistance of stainless steels under other media, nor to predicting the corrosion resistance of stainless steels to other forms of corrosion (such as pitting corrosion, uniform corrosion, stress corrosion).

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only the dated version applies to this document. For undated references, the latest edition (including all amendments) applies to this document.

GB/T 625, Chemical reagent - Sulfuric acid (GB/T 625-2007, ISO 6353-2:1983, NEQ)

GB/T 626, Chemical reagent - Nitric acid (GB/T 626-2006, ISO 6353-2:1983, NEQ)

GB/T 655, Chemical reagent - Ammonium persulfate

GB/T 665, Chemical reagent - Copper (II) sulfate pentahydrate (GB/T 665-2007, ISO 6353-2:1983, NEQ)

GB/T 2100, Corrosion-resistant steel castings for general applications (GB/T 2100-2017, ISO 11972:2015, MOD)

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cannot be ground or pickled, the surface cannot be oxidized during heat treatment.

3.1.9 During the cutting and surface grinding process of samples for Method B, Method C, Method E, Method F, Method G, the surface shall be prevented from overheating; the surface roughness Ra value of the processed sample shall generally be no more than 0.8 μ m. For samples that cannot be ground, other surface roughness can also be used according to the agreement between the two parties.

Table 1 -- Sample size and preparation requirements of Method B and Method C

Method C								
	Thickness or		Sample siz	ze	Number o	f		
Classification	diameter ^a	mm			samples		Note	
	mm	Length	Width	Thickness	pcs			
	-10	20:40	00:40		0		Take samples along the	
	≤3	30±10	20±10	-	2		rolling direction	
Steel plate, strip (flat steel)							Take samples along the	
	>3	30±10	20±10	3~4			rolling direction; process one	
							sample from one side to the	
					2		thickness of the sample;	
							process the other sample	
							from the other side to the	
							thickness of the sample	
Section							Take samples longitudinally	
steel,	-	30±10	20±10	3~4	2		from the middle of the cross	
forging							section	
	≤10	30±10	-	-	2		-	
Steel rod							Take samples longitudinally	
(steel wire)	>10	30±10	≤20	≤5	2		from the middle of the cross	
							section	
	<5	30±10	-	-	2		Take a full-tubular sample	
	5.45	00:40					Take a semi-tubular or boat-	
	5~15	30±10	-	-	2		shaped sample	
					Wall			
Seamless steel tube					thickness <	2	-	
	>15	30±10	≤20	-	4 mm			
							Process one group (2	
							samples) from the outer wall	
					Wall		to the thickness of the	
					thickness ≥	4	sample; process the other	
					4 mm		group (2 samples) from the	
						inner wall to the thickness of		
							the sample	
			L	l	l		<u> </u>	

- 1 -- welding sample;
- 2 -- welding sample;
- 3 -- welding plate.

Figure 6 -- Cross weld sampling

3.2 Sample sensitization

- **3.2.1** For ultra-low carbon stainless steels (whose carbon content is not more than 0.030%) and stabilized stainless steels (that are added titanium or niobium), when evaluating the intrinsic intergranular corrosion sensitivity, the sample shall be sensitized before the test; the sensitization system of the sample is determined through consultation between the supplier and the buyer. For austenitic stainless steels, the recommended sensitization system is 650 °C \pm 10 °C, heat preservation of 2 h, air cooling. For duplex stainless steels, the recommended sensitization system is 700 °C \pm 10 °C, 30 min, water cooling; or 650 °C \pm 10 °C, 10 min, water cooling.
- **3.2.2** For other stainless steels, whether the sample needs to be sensitized and which sensitization system is adopted shall be determined through consultation by the product standard or between the supplier and the buyer.
- **3.2.3** Welding samples are generally tested after welding. Welded parts that are subjected to hot working above 350°C after butt welding shall be sensitized after welding. The sensitization system is negotiated by both parties.
- **3.2.4** The sample sensitization shall be performed before grinding. Before sensitization and before the test, use an appropriate solvent or detergent (non-chloride) to degrease and dry the sample.

4 Method A -- 10% oxalic acid corrosion test method

4.1 Test solution

- **4.1.1** Dissolve 100 g of high-grade pure oxalic acid in accordance with GB/T 9854 in 900 mL of distilled water or deionized water, to prepare a 10% oxalic acid solution.
- **4.1.2** For the molybdenum-containing steel, when it is difficult to have a stepped structure, use 100 g of analytically pure ammonium persulfate in accordance with GB/T 655 to dissolve in 900 mL of distilled water or deionized water, to prepare a 10% ammonium persulfate solution instead of 10% oxalic acid solution.

The test report shall include the following contents:

- a) number and name of this Standard;
- b) test method;
- c) name of the sample and size of the test area;
- d) current density;
- e) etching time and temperature;
- f) metallographic photograph after etching;
- g) judgment result.

5 Method B -- 50% sulfuric acid-ferric sulfate corrosion test method

5.1 Test solution

- **5.1.1** Slowly add 236 mL of guaranteed pure sulfuric acid in accordance with GB/T 625 to a conical flask that is filled with 400 mL of distilled water to prepare a 50% (49.4% ~ 50.9%) sulfuric acid solution (pay attention to prevent bumping).
- **5.1.2** Weigh 25 g of hydrated ferric sulfate [Fe₂(SO₄)₃·xH₂O]; add about 75% (mass fraction) of ferric sulfate to the above sulfuric acid solution.
- **5.1.3** To prevent bumping, it is recommended to add debris that is made of pure aluminum oxide to the test solution.
- **5.1.4** Connect the flask to the condenser and pass cooling water; heat to boil the solution until all the ferric sulfate is dissolved.
- **5.1.5** Pay attention to eye protection and wear protective gloves during operation. Place the test flask in a fume hood.

5.2 Test instruments and equipment

- **5.2.1** It is recommended to use a 1 L ground conical flask that has a reflux condenser.
- **5.2.2** Heating device to keep the test solution slightly boiling.
- **5.2.3** Vernier caliper whose accuracy is not less than 0.02 mm;

5.3 Test conditions and procedures

W_{after} -- sample mass after the test, in grams (g);

S -- total area of the sample, in square meters (m²);

t -- test time, in hours (h).

5.5 Test report

The test report shall include the following contents:

- a) number and name of this Standard;
- b) test method;
- c) name of the sample and size area;
- d) the sensitization system shall be recorded if it is sensitized;
- e) test time;
- f) sample mass before and after the test;
- g) corrosion rate of the sample [g/(m²·h)].

6 Method C -- 65% nitric acid corrosion test method

6.1 Test solution

Use distilled water or deionized water to prepare the guaranteed pure nitric acid in accordance with GB/T 626 into 65.0% \pm 0.2% (mass fraction) nitric acid solution ($\rho_{20} = 1.40 \text{ g/mL}$).

6.2 Test instruments and equipment

Same as 5.2.

6.3 Test conditions and procedures

- **6.3.1** Measure the size of the sample; calculate the surface area of the sample (take 3 significant digits).
- **6.3.2** Weigh the sample before the test (accurate to 1 mg).
- **6.3.3** Put the sample in the test solution; use a glass holder to keep it in the middle of the solution. The amount of solution is calculated according to the surface area of the sample; its amount is not less than 20 mL/cm². Apply new test solution for every cycle. Put only one sample in each container.

- **8.3.4** Place the flask on the heating device; pass cooling water; heat the test solution to keep it slightly boiling. Continue the test for 20 h \pm 5 h. In case of dispute, 20 h shall be used.
- **8.3.5** After the test, take out the sample; wash, dry and bend.
- **8.3.6** Use new solution for each test.

8.4 Test result evaluation

- **8.4.1** The bending angle of samples is not less than 90°; the boat-shaped sample of the welded pipe is bent along the direction of the vertical welded seam; the welded joint is bent along the fusion line. For materials of low toughness, use an untested sample to determine the maximum bending angle without cracking, which is used as the bending angle of the bending test.
- **8.4.2** For press-processed parts, the diameter of the indenter for bending the sample shall not be greater than twice the thickness of the sample; for steel castings, welded pipes and welded parts, the diameter of the indenter for bending the sample shall not be greater than 4 times the thickness of the sample.
- **8.4.3** For the full-tubular sample whose diameter is not greater than 15 mm, when the flattening test is used for evaluation, the distance H between the two pressing plates is calculated according to Formula (2).
- **8.4.4** For bent samples, observe the outer surface of the curved sample for cracks due to intergranular corrosion under a 10× magnifying glass. Cracks that are generated from the edges and corners of the curved part of the sample, as well as slip lines, wrinkles, and rough surfaces that are not accompanied by cracks, cannot be considered as cracks that are caused by intergranular corrosion.
- **8.4.5** When the sample cannot be evaluated for bending or the bending crack is difficult to determine, use the metallographic method. The metallographic grinding plate shall be taken from the non-bent part of the sample (except for the welded joint and welded pipe); after etching (no over-corrosion), observe under a microscope $(150 \times \sim 500 \times)$; the allowable depth of intergranular corrosion is determined by the supplier and the buyer through consultation.

Note: If it is suspected that the crack is caused by bending, perform the same bending for a sample that has not undergone the corrosion test in the same way; after bending, perform a comparison to determine whether the crack that is seen on the corrosion test sample is caused by intergranular corrosion.

8.5 Test report

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