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Corrosion of metals and alloys — Method of oxalic acid etching test for intergranular corrosion of austenitic stainless steel

Corrosion des métaux et alliages — Méthode d'essai de gravure à l'acide oxalique pour la corrosion intergranulaire de l'acier inoxydable austénitique

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Foreword

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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).

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For an explanation of 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 www.iso.org/iso/foreword.html.

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Intergranular corrosion (IGC) is a form of localized corrosion which occurs along grain boundaries. IGC of stainless steel is attributable to grain boundary segregation and precipitates. When the formation of chromium-rich grain boundary carbides occurs, it leads to continuous chromium depletion adjacent to the grain boundary, and IGC occurs in certain corrosive media. This process is usually known as sensitization. Sensitization of stainless steel can be evaluated by ISO IGC tests for austenitic stainless steels: such as ISO 3651-1, ISO 3651-2, and ISO 12732. On the other hand, oxalic etching tests (JIS G0571:2003 and ASTM A262-15 Practice A) cannot evaluate sensitization, however, they can evaluate non-sensitized condition of austenitic stainless steels and suspicions of sensitization by observing the etched surface of the stainless steels. This is because it does not etch chromium depletion adjacent to the chromium-rich grain boundary precipitates but chromium-rich grain boundary carbides themselves. If sensitization is suspected, IGC tests, ferric sulfate-sulfuric acid test (see ISO 3651-2), 65 % nitric acid test (see ISO 3651-1), copper sulfate-sulfuric acid test (see ISO 3651-2) or EPR test (ISO 12732) should be conducted to evaluate sensitization of austenitic stainless steels.

The main benefits of these oxalic acid etching tests are that they are simple, easy and rapid. This document is based on JIS G0571:2003.

Corrosion of metals and alloys — Method of oxalic acid etching test for intergranular corrosion of austenitic stainless steel

1 Scope

This document specifies a method to test for intergranular corrosion (IGC) in austenitic stainless steels using an oxalic acid etch. This method can evaluate non-sensitization of austenitic stainless steels by observing the structure of the etched surface with a microscope after performing electrolytic etching (hereinafter called "etching") on austenitic stainless steel in an oxalic acid solution.

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 3651-1, *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, *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*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 8044, *Corrosion of metals and alloys — Vocabulary*

ISO 12732, *Corrosion of metals and alloys — Electrochemical potentiokinetic reactivation measurement using the double loop method (based on Cihal's method)*

3 Terms and definitions

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

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

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

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

3.1
sensitization
effect coming when austenitic stainless steels are slowly cooled through the range 550 °C to 850 °C or heated in the range 550 °C to 850 °C for more than a certain time, they frequently become susceptible to IGC in a corrosive medium

3.2
stabilized austenitic stainless steel
austenitic stainless steel that contains stabilizers such as titanium or niobium, which form carbides that are more stable than chromium carbides

4 General principles

Observing the structure of the etched surface with a microscope after performing etching on austenitic stainless steel in an oxalic acid solution will determine whether or not ISO 3651-1, ISO 3651-2, or ISO 12732 shall be used.

5 Apparatus

5.1 DC supply, a variable resistor and an ammeter should be used to control the DC power supply, which can supply a sufficient amount of current for etching the test piece. For example, a DC power supply with rated output voltage 15 V and rated current output 20 A, would be suitable.

5.2 Cathode, an austenitic stainless steel beaker – or a piece of austenitic stainless steel with sufficient area – should be used as a cathode.

5.3 Holder, a properly shaped electrical holder should be used to retain the test piece as an anode in the test solution.

5.4 Test vessel should be made of an inert material. For example, corrosion resistant alloys such as an austenitic stainless steel, may also be used as a cathode.

5.5 Thermometer, capable of measuring the temperature of the test solution with an accuracy of ±1 °C should be used.

6 Sensitization heat treatment for test specimens

A sensitization heat treatment for the test specimens should be applied for the ultralow carbon austenitic stainless steel type (carbon mass fraction <0,030 %) and stabilized austenitic stainless steel. The heat treatment shall be performed before polishing. As an example, the specimen could be heated for 30 min at a temperature of 700 °C ± 10 °C, and then cooled with water. And the specimen can be heated for 10 min at a temperature of 650 °C ± 10 °C, and then cooled with water. The sensitization heat treatment may be altered with the agreement of relevant parties.

7 Test specimens

7.1 The test specimen should be arranged so that the etched surface can be examined with a microscope. When a welded part is included, its section shall include regions of base material, heat-affected zone and weld material. If the specimen has a complex shape, the direction of the surface to be examined with a microscope may be altered according to the agreement between the relevant parties.

7.2 Typically, a saw can be used to cut specimens, although other methods such as guillotine or water-jet cutting can be used if available.

7.3 The etched surface to be examined with a microscope shall be finely polished by buffing (for example by polishing with a diamond particle suspension) prior to etching and examination.

7.4 Parts other than the etched surface to be examined with a microscope shall be isolated from the test solution with an insulating material. Paraffin wax, plastic tape, resin or paint can be used as insulating material.

8 Test solution

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

Dissolve 100 g of oxalic acid crystals ($H_2C_2O_4 \cdot 2H_2O$) in 900 ml of distilled water or deionized water, ISO 3696:1987, Grade 3, and stir until all crystals are dissolved.

In the case of molybdenum-containing steel types, in which the step structure is difficult to generate, an ammonium peroxodisulfate [$(NH_4)_2S_2O_8$] solution can be used instead of an oxalic acid solution. For the solution in this case, dissolve 100 g of ammonium peroxodisulfate in 900 ml of distilled or deionized water and stir until all the crystals are dissolved.

9 Test procedure

The test procedure shall be as follows:

a) The temperature of the test solution shall be 20 °C to 50 °C.

   In the case of the test solution temperature over 50 °C, a usable etched surface cannot be obtained.

b) The polished surface by buffing of the test piece shall be placed as an anode in the oxalic acid test solution. Etching shall be performed for 90 s at a current density of 1 A/cm^2.

   In the case of using the ammonium peroxodisulfate solution, etching shall be performed for 5 min to 10 min at a current density of 1 A/cm^2.

c) After etching is performed, the test piece shall be removed from the test solution, washed with running water and dried. The entire area of the etched surface shall be observed with a microscope to determine the classification of the etching structure based on Table 1 and Figures 1 to 3. Furthermore, the microscopic magnification shall be 200× to 500× for rolled, forged products, cast steel products, welded areas or similar.

Table 1 — Classification showing the crystal grain boundary condition

Etching structure		Explanation
Symbol	Name	
A	Step structure	This structure appears because the corrosion rate differs depending on the crystal orientation (see Figure 1).
B	Dual structure	Grooves are formed at the crystal grain boundaries. However, there are no crystal grains completely surrounded by grooves (see Figure 2).
C	Groove structure	There are one or more crystal grains completely surrounded by grooves (see Figure 3).

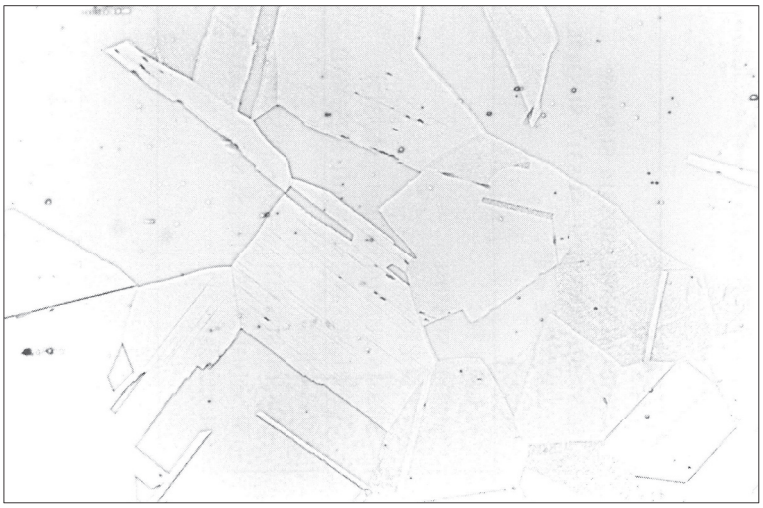

Figure 1 — Step structure (symbol A)

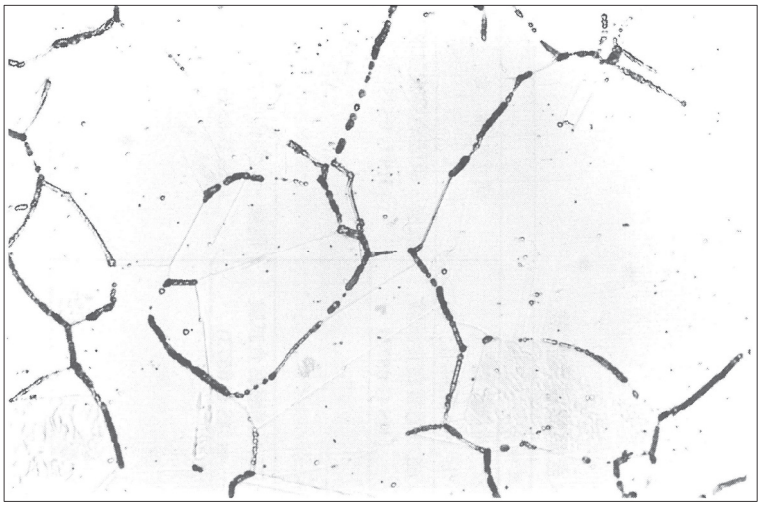

Figure 2 — Dual structure (symbol B)

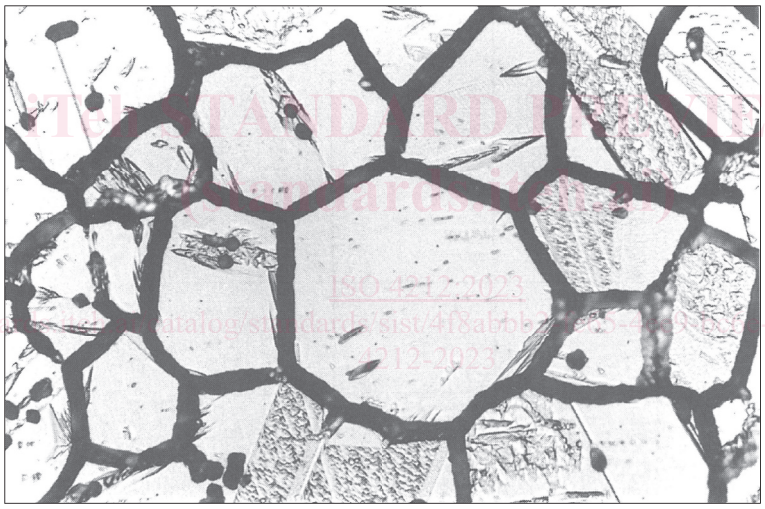

Figure 3 — Grooved structure (symbol C)

10 Evaluation of etching structure

Specimens having a step structure or dual structure need not be subjected to the ferric sulfate-sulfuric acid test (see ISO 3651-2), 65 % nitric acid test (see ISO 3651-1), copper sulfate-sulfuric acid test (see ISO 3651-2) or EPR test (see ISO 12732). Specimens having the grooved structure are suspected of sensitization and should be subjected to the ferric sulfate-sulfuric acid test (see ISO 3651-2), 65 % nitric acid test (see ISO 3651-1), copper sulfate-sulfuric acid test (see ISO 3651-2) or EPR test (see ISO 12732) in order to evaluate sensitization of the specimens.

11 Test report

The test report shall include the following information:

a) a reference to this document, (i.e. ISO 4212:2023);