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Corrosion of metals and alloys — Method of measuring the pitting potential for stainless steels by potentiodynamic control in sodium chloride solution

Corrosion des métaux et alliages — Méthode de mesure du potentiel de piqûre des aciers inoxydables par contrôle potentiodynamique en solution de chlorure de sodium

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The committee responsible for this document is ISO/TC 156, *Corrosion of metals and alloys.*

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Introduction

Although stainless steel is generally used as a corrosion-resistant material, it is susceptible to pitting corrosion, crevice corrosion, stress corrosion cracking, etc. Among those, pitting corrosion is one of the most common phenomena that occur on stainless steels. A commonly used parameter to evaluate the pitting corrosion resistance of stainless steel, is so-called pitting potential that defines the lowest potential below which stables pits are not considered to grow. Since pitting corrosion generally shows a stochastic nature dependent upon inhomogeneity in terms of size, orientation, alloying components, impurity, inclusions, segregation, surface treatment, history, elapsed time, fluctuation of environment, etc., its measurement requires at least a couple of values.

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Corrosion of metals and alloys — Method of measuring the pitting potential for stainless steels by potentiodynamic control in sodium chloride solution

1 Scope

This International Standard describes the procedure for determining the pitting potential for stainless steels (austenitic, ferritic/austenitic, ferritic, martensitic stainless steel) under potentiodynamic control.

The principal advantage compared with other potentiostatic test methods[1][2] is the rapidity of this test method, with which the pitting potential can be measured in a single potential scan.

The pitting potential as determined by this International Standard can be used as a relative index of performance. For example, one can compare the relative performances for different lots of stainless-steel grades and products. The test described in this International Standard is not intended to determine the pitting potential at which actual pitting can occur under real service conditions, or not.

2 Principle

The test involves increasing the anodic potential of the specimen at a specified scan rate while exposing the specimen to a normalized sodium chloride solution at a constant temperature.

The pitting potential (V'_{c10} or V'_{c100}) (see JIS G 0577[3]) is defined as the potential at which the current density exceeds 10 μA/cm^2 or 100 μA/cm^2 for more than 60 s. A 60 s delay is used in order to ensure that the observed current increase originates from stably propagating pitting, since short-lived current peaks originate from metastable pitting.

The specimen holder and the specimen itself are designed to ensure that crevice corrosion does not occur.

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3 Apparatus

3.1 Potentiostat

The potentiostat shall be capable of controlling the electrode potential to within ±1 mV of a preset value.

3.2 Electrode potential-measuring instrument

The instrument shall have high input impedance sufficient to eliminate potential read error due to current drawn by the instrument during measurement; impedance of the order of 10^{11} Ω to 10^{14} Ω is typical. The sensitivity and accuracy of the instrument should be sufficient to detect a change of 1,0 mV.

3.3 Current-measuring instruments

The current in the circuit is evaluated from the potential drop measured across a known resistor. In many potentiostats, this measurement is made internally. But measurements may also be made externally by locating a resistor in the current line from the auxiliary electrode to the auxiliary connection of the potentiostat. The instrument shall be capable of measuring a current within 2 % error around the actual value.

3.4 Specimen holder

3.4.1 Any part of the specimen holder coming into contact with the test solution shall be made of an inert material.

3.4.2 The specimen holder shall be designed to ensure that crevice corrosion does not occur at the contact area between the specimen holder and the specimen. Some methods to prevent such crevice attack, using a flushed port cell, or using a flushed specimen holder, are outlined in A.1 and A.2.

3.4.3 If the specimen holder avoiding crevice corrosion cannot be used, the specimen electrode could be treated by the special method as outlined in A.3.

3.5 Test cell

3.5.1 The test cell shall contain the test specimen, a Luggin capillary probe connected to an external reference electrode for measuring the electrode potential, an auxiliary electrode, a port for insertion of a temperature measuring device, a bubbler for de-aeration by inert gas such as N_2 or Ar, and/or a facility for stirring the solution in a repeatable manner.

NOTE This can be achieved by using a mechanical stirring device, or simply by bubbling inert gas through the solution at a controlled rate.

An accuracy of the temperature measuring instrument shall be ±0,4 °C.

3.5.2 A double-walled cell is commonly used to enable the solution to be cooled or to be heated by recirculating a liquid from an external cooling or heating bath to the outer chamber of the cell.

3.5.3 The tip of the Luggin capillary probe shall be positioned so that it is at a distance from the specimen of about, but not closer than, twice the diameter of the tip.

3.5.4 Any part of the test cell or specimen holder that comes into contact with the solution shall be constructed from an inert material. Polycarbonate, glass and polytetrafluoroethylene (PTFE) are suitable materials.

3.5.5 The ratio of the volume of solution in the test cell to the specimen area shall be at least 100 ml/cm^2.

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3.6 Auxiliary electrode

The auxiliary electrode is commonly prepared from high purity platinum. Other materials may be used provided they are inert. The auxiliary electrode may be constructed in the form of sheet, rod, wire, or in the form of gauze supported on a glass frame. The area of the auxiliary electrode shall be at least the area of the specimen.

Graphite may be used as an auxiliary electrode, but care shall be taken to avoid contamination; desorption of species retained in the graphite may be necessary prior to usage.

3.7 Reference electrode

3.7.1 The reference electrode shall be maintained at ambient temperature external to the test cell, and shall be connected to the test cell via a Luggin capillary probe.

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3.7.2 Commonly used electrode is the saturated silver/silver chloride electrode (sat.SSCE). The potentials of these electrodes relative to the standard hydrogen electrode at 25 °C are given in ISO 17474.[4]

If the saturated calomel electrode (SCE) is used, strict control to handle mercury and mercury containing substances in terms of health and environmental issues shall be taken. The use of saturated calomel electrode shall follow nation's rules and regulations.

4 Specimens

4.1 The specimen shall be taken from a test material such that the test area is minimum 1,0 cm^2. Different surfaces are permitted, if they are used in actual application: e.g. different heat treatments, and different surface finish.

4.2 The specimen may be taken by sawing, cutting, grinding, etc. The depth affected by machining shall be removed by progressive grinding so that the traces of machining may not affect the test results.

NOTE If relevant parties agree, any surface finishes different from the above recommendation may apply as far as they are reproducible.

5 Procedure

5.1 Preparation of reference electrodes

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5.1.1 The difference in potential among the reference electrode and two other validation electrodes shall be measured. The latter electrodes shall be traceable to the standard hydrogen electrode, and shall be maintained solely for the purpose of validation. If the potential is different by greater than 3 mV, the electrode shall be rejected.

5.1.2 The validation electrodes shall be stored in optimum conditions and regularly compared. If the potential difference between these varies by more than 1 mV, replacement shall be undertaken.

5.2 Preparation of specimen

5.2.1 The final grinding of the specimen may be dry or wet. Before measurement, the specimen is recommended to be ground with 600 grit paper, and shall then be thoroughly cleaned.

NOTE 1 After grinding, it takes time for the air formed film to achieve a quasi-steady condition. The most rapid change in filming occurs in the first period with progressive stabilization at longer periods.

NOTE 2 A minimum time period of 24 h is recommended. However, a shorter period may be adopted depending on the purpose of the test. Nevertheless, the time period in a set of tests should be consistent.

NOTE 3 If relevant parties agree, surface finishes different from the above recommendation may apply as far as they are reproducible.

5.2.2 The specimen shall be cleaned prior to immersion into the test solution, by degreasing, rinsing in high purity water (with a conductivity less than 1 µS/cm), followed by ethanol or a similar solvent, and then air drying. After degreasing, care shall be taken not to contaminate the test surface of the specimen.

5.3 Preparation of solution

5.3.1 The solution shall be prepared using reagent-grade chemicals and high purity water.

5.3.2 The test solution should reflect the intended application; otherwise 1 M (k mol m^{-3}) sodium chloride aqueous solution may be used.

NOTE The recommended test solution of 1 M sodium chloride aqueous solution can be prepared by dissolving 58,44 g of reagent-grade sodium chloride into 1 000 mL of distilled or ion-exchange water.

5.4 Setting up test

5.4.1 The exposed surface area of the test specimen shall be measured.

5.4.2 The standard temperature of the test solution shall be (30 ± 1) °C.

The tests may be performed at a different temperature depending on the purpose, but the same temperature for a particular set of tests shall be applied.

5.4.3 For anodic polarization, completely immerse the test surface area in the de-aerated test solution; leave it to stand there to stabilize temperature and corrosion potential for at least 1 min. Then, start potential sweeping at the rate of 10 mV/min from corrosion potential until the anodic current density reaches more than 500 µA/cm^2 and less than 1 000 µA/cm^2. In case statistical analysis of the potential data was intended, the sweep rate of 20 mV/min may be used. If these sweep rates are not available by reason of the apparatus or other, a sweep rate close to it may be used.

Regardless of the type of test assembly (see Annex A), deaeration of the solution shall be undertaken prior to immersion. A deaeration period of 1 h per litre of test solution is usually sufficient at typical gas flow rates of 0,1 L/min of e.g. N_2 or Ar.

5.4.4 The pitting potential shall be expressed by the value of the potential for which pitting becomes stable; i.e. current density increases continuously with potential as shown in Figure 1. The pitting potential is defined as the potential corresponding to the anodic current density of 10 µA/cm^2 or 100 µA/cm^2 of the stable pitting region. The pitting potentials shall be denoted as V'_{C10} or V'_{C100}, respectively. Figure 1 shows an example of the measurement.

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5.4.5 For each test, use a fresh specimen and a fresh test solution.

5.5 Recording

The value of pitting potential measurement shall be recorded in volts (V), down to three decimal places. Also, the sweep rate and grinding condition prior to the test, the reference electrode, time elapsed between grinding and immersion, and the test temperature shall be noted (see Clause 7).

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