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Corrosion of metals and alloys — Measurement of critical crevice temperature for cylindrical crevice geometries in ferric chloride solution

Corrosion des métaux et alliages — Mesure de la température critique de corrosion caverneuse de crevasses de géométries cylindriques dans une solution de chlorure ferrique

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

Introduction

Various dimensions of pipes and tubes are used in industries such as nuclear power plants, thermal power plants, desalination plants, oil refining facilities and chemical plants, etc. Materials evaluation for these pipes, tubes and fittings makes all the difference to safety and life cycles in these industrial facilities.

There are numerous standards that specify test methods and corrosion resistance for pitting and crevice corrosion. Many of them address the corrosion rate or critical pitting temperature (CPT) or critical crevice temperature (CCT) of plate type specimens, but they are not concerned with other geometries and relations of corrosion resistance between geometries.

It is important that the initiation temperature of crevice corrosion be measured in cylindrical crevice geometries for high corrosion resistant alloys, including from traditional stainless steels (300 series) to super stainless steels (higher alloyed stainless steels).

This document provides the test method that measures crevice corrosion resistance in crevice geometries such as tube/tube sheet of heat exchangers and pipes/pipe supports, flange/couplings, bolts/nuts, etc. in industrial facilities. It can be used as a guideline which offers the criterion for materials selection of such components.

Most crevice corrosion tests are performed conforming to some standards, but these test methods use a flat geometry of an artificial crevice which shows different critical crevice temperatures to those of cylindrical crevice geometry. Therefore, results of these tests have created controversies in related industries. It has been found that the identical CCT between plate type and cylindrical type specimens could be achieved with an appropriate torque applied to the cylindrical specimen.

Corrosion of metals and alloys — Measurement of critical crevice temperature for cylindrical crevice geometries in ferric chloride solution

1 Scope

This document specifies a methodology for ranking the crevice corrosion resistance of stainless steels and related alloys when exposed to oxidizing chloride solution. This document allows the measurement of critical crevice temperatures of tube/rod type specimens equal to those of plate type ones made up of the same material by chemical initiation of crevice corrosion, but not by the electrochemical method of ISO 18070. The test method in this document defines the apparatus and the procedure used to measure the temperature of crevice corrosion initiated in pipes and tubes using cylindrical specimens. This method has also been proved to apply to plate type specimens.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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 and the following apply.

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

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

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

3.1
critical crevice temperature
CCT
minimum temperature required to produce a crevice attack of at least 0,025 mm corroded depth on the bold surface of a specimen beneath the *crevice former* (3.2), with the edge attack ignored

Note 1 to entry: Measured in degrees Celsius.

3.2
crevice former
component of the crevice corrosion test tool assembly, which allows crevice corrosion to be induced in a contacted test specimen by applying certain torques

4 General principles

4.1 Objective

This document describes laboratory tests for measuring CCT of stainless steels and high corrosion resistant alloys with cylindrical (including circular arc) and flat plate geometries in oxidizing chloride solutions. Cylindrical crevice geometries emulate crevices frequently coming from tube/tube sheet of heat exchangers and pipes/pipe supports, flange/couplings, bolts/nuts, etc. in industrial facilities. There have been differences of CCT between plate type and cylindrical type specimens of same materials which require measuring CCT of a part of real pipes and tubes in service or in fabrication. This document aims to rank the crevice corrosion resistance of stainless steels and related alloys, eliminating the geometric (tubular or rod or plate) and size (radius of curvature) effect, when exposed to oxidizing chloride solution.

4.2 Immersion test

4.2.1 General

Chloride containing solutions such as ferric chloride and acidified ferric chloride solutions are useful to determine localized corrosion rate. This test method uses 6 mass % ferric chloride or 6 mass % ferric chloride solution +1 volume % HCl solution. A crevice former is essential to produce crevice corrosion and its dimension shall be modified according to various pipes and tubes. Artificial crevice formers with a specimen shall be completely immersed in the test cell. The volume of solution shall be at least 500 ml/dm^2 of specimen surface area.

4.2.2 CCT measurements in single test cell

A critical crevice temperature, which shows the initiation of crevice corrosion, shall be measured for a specimen from a certain temperature. After 24 h of immersion, the specimen is removed for inspection of crevice corrosion. If there is no initiation of crevice corrosion, then a different specimen shall be subjected to the test with a 5 °C increase from the previous temperature. The test shall be repeated with increases of 5 °C until crevice corrosion initiates. Every specimen at each temperature shall be fabricated from the same product or from the identical heat of a raw material with the same geometry.

4.2.3 CCT measurements in multiple test cells

Tests in 4.2.2 can be performed simultaneously in multiple test cells in which each specimen is immersed in a different temperature for speed tests. Each specimen shall be fabricated from the same product or from the identical heat of a raw material with the same geometry. Applied torque condition for each specimen shall be same if the specimens have the same radius of curvature.

4.3 Criterion for CCT

The ferric chloride solution used in this document is a strong oxidizing and corrosive medium causing pitting or crevice corrosion in high corrosion resistant alloys. Even though measuring the weight of a specimen before and after test makes it possible to show the initiation of crevice corrosion, credibility of the results is not high because the strong test solution may inadvertently corrode the edges of the specimen. The criterion for CCT, as described in 3.1, is the lowest temperature for which a corroded depth of at least 0,025 mm shows on the surface of the specimen beneath the crevice former, with the edge attack ignored.

5 Apparatus

5.1 Crevice former assembly

As shown in Figure 1, the frame of the crevice test assembly is made up of titanium that has a very high corrosion resistance against pitting or crevice corrosion. Crevices shall be formed with fittings, as shown in Figures 2 to 5, to prevent galvanic corrosion when a specimen makes contact with the titanium frame.

Locate the left and right polytetrafluoroethylene (PTFE) block decks in advance, on which a cylindrical specimen is spanned and held as shown in Figure 1. Make a position of lower crevice former between them, which has a same curvature with a specimen.

The curvature of lower crevice former for a part of circular arc geometry (e.g. of big size specimen) should be convex upwards.

CCT of the plate type specimen shall be measured with a flat crevice former.

Mount a specimen on the lower crevice former, then add an upper crevice former on top of it. Install a titanium washer on the upper crevice former; this evenly distributes the stress on the surface of the specimen when a force is added with a titanium bolt contacted on top of the washer. Finally, wrench the titanium bolt up to the determined amount of torque with a torque wrench.

The requirements for materials and dimensions of critical crevice temperature test assembly are as follows:

— materials of assembly: titanium grade 2 or equivalent;

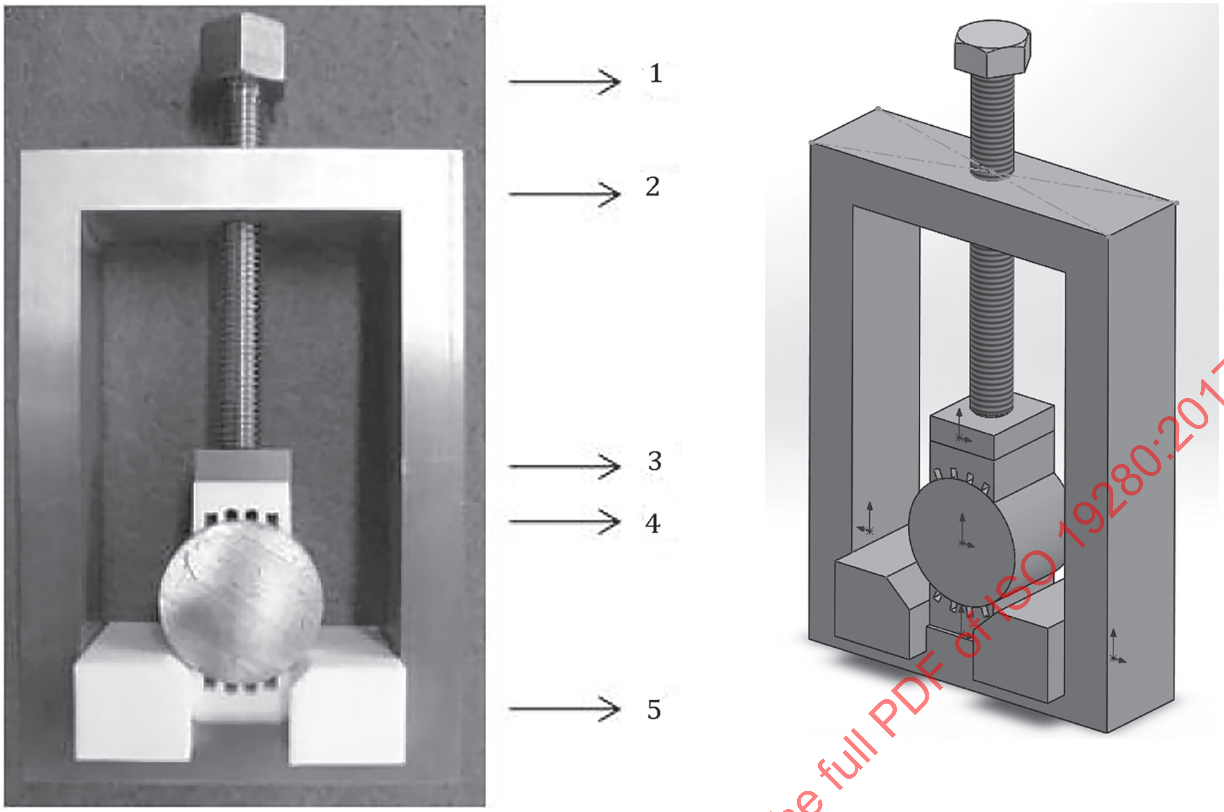
— dimension of assembly: various dimensions fit for cylindrical products;

— artificial crevice former: machined PTFE upper and lower blocks rounded surface with grooves for contacting to a specimen;

— supporting decks: right and left PTFE blocks with rounded surface for a specimen to be clamped inside the assembly;

— crevice former; PTFE crevice former should meet the mechanical requirement which the tensile strength is higher than 14,5 MPa.

— titanium bolt (see ISO 4017; thread size M8) for applying torque to the specimens.



a) Front view photo

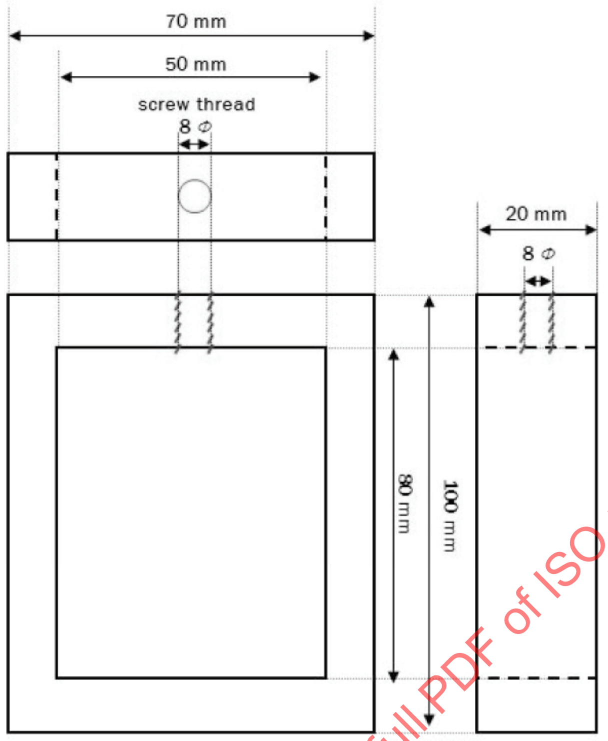
b) Side view diagram

Key

1 Ti bolt
2 Ti frame
3 Ti washer
4 crevice washer
5 deck block

Figure 1 — Assembled crevice test apparatus for cylindrical specimen

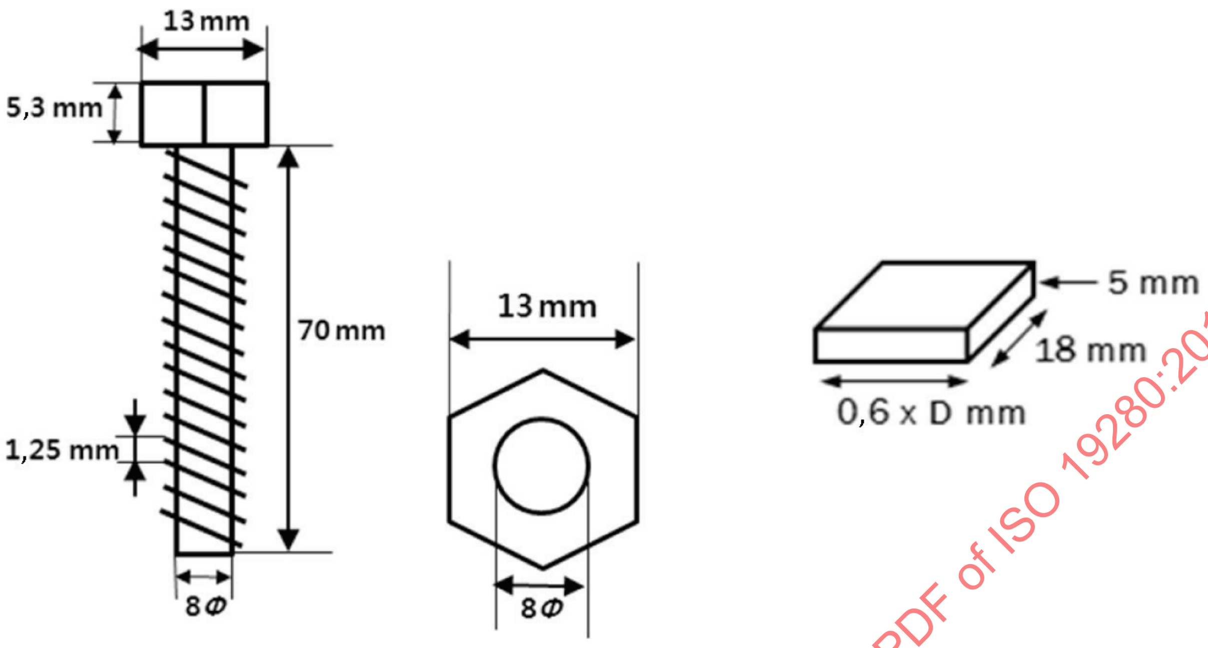
Dimensions in millimetres



NOTE: Crevice corrosion test frame dimensions, in millimetres:

Outside		Inside		Thickness
Length	Width	Length	Width	
100	70	80	50	20

Figure 2 — Ti frame of assembled crevice test apparatus for a specimen of benchmark scale



a) Ti bolt (with hexagon bolt head: ISO 4017**)** **b) Ti washer**

NOTE 1: Ti bolt and dimensions of Ti bolt and Ti washer, in millimetres:

Components	Width	Thickness	Length	Pitch	Screw diameter
Ti bolt	13 (head)	5,3 (head)	70	1,25	8 (out diameter)
Ti washer	0,6 x D	5	18	—	—

NOTE 2 D = diameter of cylindrical specimen.

Figure 3 — Ti Bolt and Ti Washer of assembled crevice test apparatus for a specimen of benchmark scale

5.2 Test cell

5.2.1 General

Either wide mouth flasks or suitably sized test beakers may be used. Condensers shall be used for the tests at elevated temperatures in which solution evaporation may occur.

5.2.2 Flask requirements

The volume of the wide mouth flask or beaker used shall be of 1 000 ml. Tall form flasks can be used. The flask shall have a diameter of about 80 mm and a height of about 120 mm to allow the immersion of the assembled test apparatus.

Dimensions designated in this document are meant for a cylindrical specimen with a surface area of 30 cm^2 maximum provided that the titanium frame of the test assembly has been passivated. Immersion condition of at least 5 ml per cm^2 of specimen surface area (4.1) is the minimum requirement, not considering the surface area of the whole test assembly.

The dimension of the container and the amount of solution for bigger specimens (> 30 cm^2) shall be decided according to the surface area of the specimen and the dimension of the test assembly in proportion to the minimum 5 ml/cm^2 ratio.

5.2.3 Condenser

A variety of condensers may be used in conjunction with the flasks described in 5.2.2. These include the cold finger-type or Allihn type condensers having straight tube ends or tapered ground joints. Straight end condensers can be inserted through a bored rubber stopper. Likewise, a simple U tube condenser can be used.

A fume hood and venting shall always be used due to the harmful gas and possible build-up of gas pressure that may result from the corrosion process.

5.3 Test temperature

The thermostat bath shall have the capability of providing a constant temperature between 0 °C ± 1 °C and 85 °C ± 1 °C.

5.4 Crevice assembly

5.4.1 Supporting deck blocks and crevice formers

Lower supporting deck blocks are composed of two parts: left deck and right deck. The material of each deck is PTFE block.

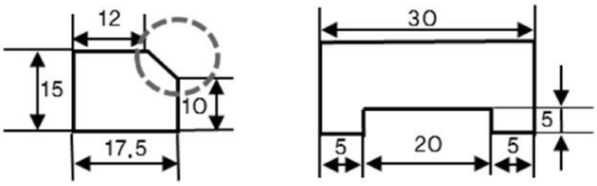
The dimension is varied according to that of a loaded cylindrical specimen. The dimensions shown in Figure 4 are relative to the decks for a cylindrical specimen and a plate specimen.

The dashed circle in Figure 4 a) is expressed as flat face, but it shall be rounded according to the radius of a loaded cylindrical specimen being kept from contacting the specimens. The crevice assembly, consisting of solid PTFE segmented crevice former blocks, each having a number of grooves and plateaus, shall be used.

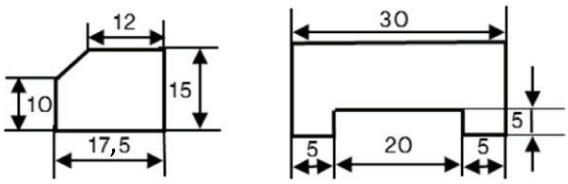
The crevice former design shown in Figure 5 is one of a number of variations for the various diameters of cylindrical (including circular arc geometry) specimens.

The dimensions of supporting deck blocks and crevice formers for a bigger specimen (Ø > 25 mm) shall be decided in proportion to its dimension rate compared to the benchmark dimension of 5.4.1.

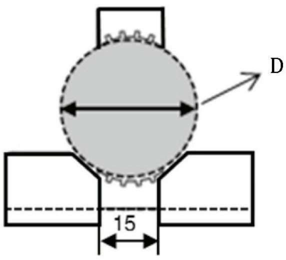
Dimensions in millimetres



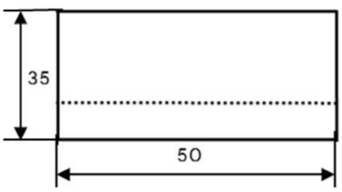
a) Left deck for a cylindrical specimen



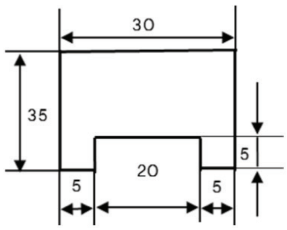
b) Right deck for a cylindrical specimen



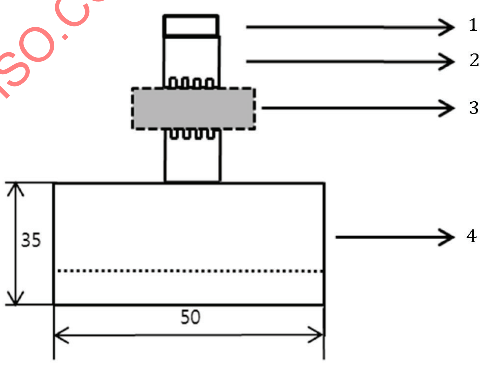
c) Cylindrical specimen mounted on the deck blocks



d) Front view of deck block for plate specimen



e) Side view of deck block for plate specimen



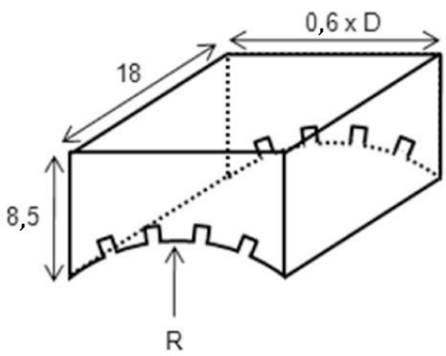
f) Left deck for a cylindrical specimen

Key

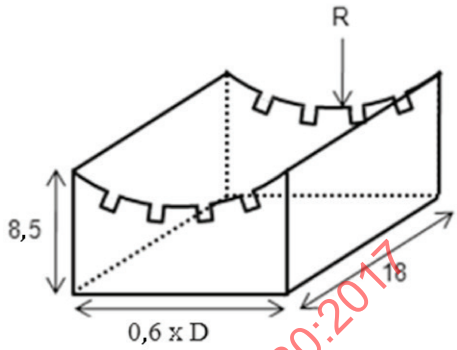
D tube diameter
1 Ti washer
2 crevice former
3 test specimen
4 deck block

Figure 4 — PTFE-supporting deck blocks: example for cylindrical specimen and plate specimen

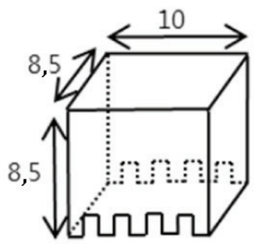
Dimensions in millimetres



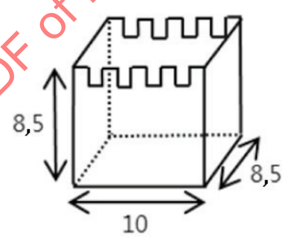
a) Upper part for a cylindrical specimen



b) Lower part for a cylindrical specimen



c) Upper part for a plate specimen



d) Lower part for a plate specimen

NOTE 1:

Dimension of crevice former for cylindrical specimen, in millimetres					
Upper part		Lower part		Thickness	Number of slots, min.
Length	Width	Length	Width		
18	0,6 × D	18	0,6 × D	8,5	4
Dimension of crevice former for plate type specimen, in millimetres					
Upper part		Lower part		Thickness	Number of slots, min.
Length	Width	Length	Width		
8,5	10	8,5	10	8,5	4

NOTE 2 The radius of crevice former, R, shall be modified as the diameter of cylindrical specimen, and D is the diameter of cylindrical specimen. In order to measure crevice corrosion of the plate, use the flat-plate.

NOTE 3 The width and depth of the slots shall be at least 1 mm, respectively.

NOTE 4 The width of crevice former cylindrical specimen shall be modified depending on the diameter, D, of the specimen; 0,6 x D is recommended.

NOTE 5 When the torque is applied for the plate specimen, it should be prepared not to be twist.

Figure 5 — PTFE crevice former

5.4.2 Reuse of crevice assemblies

After being subjected to the specified torque, the PTFE segmented crevice former of Figure 5 shall not be reused. The artificial crevice former shall be replaced with new one in every test performed. The supporting block decks can be reused provided they have not become deformed during testing. Before reuse, each washer shall be inspected for evidence of distortion and other damages. If impaired, they shall be discarded. In some cases, the supporting block decks may become stained with corrosion products from the tested alloy. Generally, these specks can be removed by immersion in dilute HCl (for example, a volume fraction of 5 % to 10 %) at room temperature, followed by brushing with mild detergent and thorough rinsing with water.

5.4.3 Bolt and washer

Commercially pure titanium (grade 2 or equivalent) is required for each assembly. Each assembly comprises a threaded bolt and a clamp for supporting structures. The bolt length shall be sized as shown in Figure 3 and described in 5.1.

5.5 Required apparatus

The apparatus required is as follows:

— a torque limiting nut driver (torque wrench), for assembly of the crevice test specimen;

— a microscope (for example, 20 × magnification), for crevice corrosion detection;

— a 3D microscope or confocal microscope, for determining the depth of crevice corrosion;

— an electronic balance (optional), for accurately recording the mass loss;

— a camera, for making a photographic record of the mode and extent of crevice corrosion.

6 Test solutions

For the test, two ferric chloride solutions can be used.

a) Dissolve 68,72 g (reagent grade) of ferric chloride, $FeCl_3 \cdot 6\ H_2O$ in 600 ml of distilled water. This will produce a solution containing about a mass fraction of 6 % $FeCl_3$.

b) Dissolve 68,72 g (reagent grade) of ferric chloride, $FeCl_3 \cdot 6\ H_2O$ in 600 ml of distilled water and add 16 ml of reagent grade concentrated 36,5 % to 38,0 % hydrochloric acid (HCl). This will produce a solution containing about 6 mass % $FeCl_3$ and 1 volume % HCl resulting in a pH controlled environment over the test temperatures.

The volume of test solution and reagents for a bigger specimen (Ø > 25 mm) shall be decided in proportion to its dimension rate referring to the benchmark solution of Clause 6.

7 Test specimen

7.1 Specimen size

A specimen of 20 mm length is recommended as a standard size, although various diameter and thickness of tubes or rods or plates can be tested by this method. Measure the dimensions of the specimen, including diameter, thickness, etc., and calculate the total exposed area to establish the initial surface area and volume.

7.2 Surface finish

The surface finish shall reflect the intended application. Alternatively, for comparative purposes, a 120-grit abrasive paper has been found to provide a satisfactory standard finish. Wet grinding is preferred, but if dry grinding is used it shall be performed slowly to avoid overheating. The sharp edges of a specimen shall be deburred by use of fine-grit grinding paper to avoid edge effect of corrosion. Any pollutants or foreign substances shall be removed and cleaned not to affect the evaluation.

Clean the specimen surface after grinding it with the 120-grit abrasive paper, rinse well with water followed by alcohol and acetone or similar solvent and dry in air. After cleaning the specimen should be handled with care to avoid any grease marks. Weigh each specimen to the nearest 0,001 g or better and store them in a desiccator until ready for use. The specimen shall be exposed to 24 h air passivation before test.

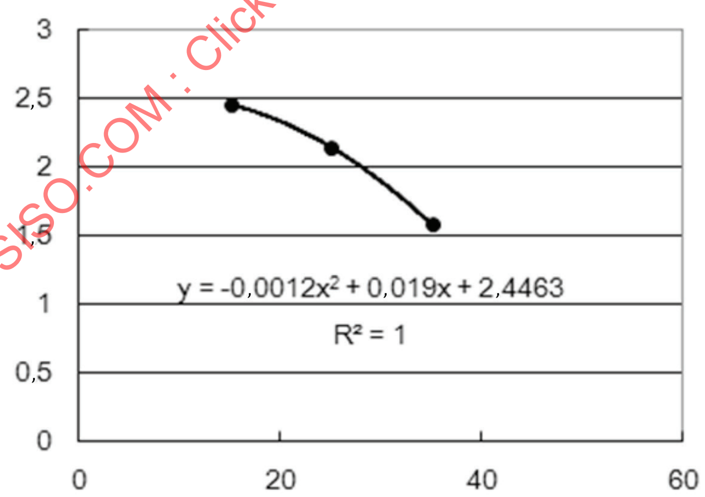
8 Experimental procedure

Apply the artificial crevice formers to the specimen. A PTFE crevice former and decks, and a bolt and washer of titanium (grade 2) shall be used to fasten the crevice formers to the specimen, as illustrated Figure 1. A torque shall be applied using a drive torque limiting nut-driver (torque wrench). The applied torque shall be modified depending on the radius of curvature of the specimen. The recommended torques for cylindrical specimens of 15 mm, 25 mm and 35 mm in diameter (or equivalent radius of curvature) are of 2,45 Nm, 2,14 Nm and 1,58 Nm respectively.

The torque to apply (in Nm) can be calculated from Formula (1) and from the graph given in Figure 6.

$$\text{Applied torque, Nm} = -0{,}00\,12D^2 + 0{,}019D + 2{,}446\,3 \tag{1}$$

However, this method cannot be used for specimens of less than 15 mm in diameter, which need such high torques that it may lead to the deformation of the PTFE crevice formers.



Key

X diameter, in millimetres

Y torque, in Newton metres

Figure 6 — Relationship between tube diameter and applied torque for CCT test

Pour an appropriate amount of solution in the test cell as described in Clause 6. Close the cell with a glass cover and install the water condenser, set and maintain the test temperature of interest. Verify the temperature before starting the test.