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Corrosion of metals and alloys — Uniaxial constant-load test method for evaluating susceptibility of metals and alloys to stress corrosion cracking in high-purity water at high temperatures

Corrosion des métaux et alliages — Méthode d'essai sous charge uniaxiale pour l'évaluation de la sensibilité des métaux et des alliages à la fissuration par corrosion sous contrainte dans l'eau de haute pureté à hautes températures

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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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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.

Corrosion of metals and alloys — Uniaxial constant-load test method for evaluating susceptibility of metals and alloys to stress corrosion cracking in high-purity water at high temperatures

WARNING — This document can involve hazardous materials, operations and equipment. It is the responsibility of the user of this document to consult and establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

1 Scope

The document specifies a method for undertaking uniaxial constant load testing of the susceptibility of a metal, or an alloy, to stress corrosion cracking (SCC) in high-purity water environments at high temperature (above the boiling point of water at normal pressures) and pressure. The test method is particularly applicable to simulated primary water environments of light water reactors (LWRs).

The test method enables assessment of the relative resistance to SCC of a material in different environments and the comparative resistance of different materials (using the same environment, specimen dimensions and loading).

The terms “metal” and “alloy”, as used in the document, include weld metals and weld heat affected zones.

2 Normative reference

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 7539-1, *Corrosion of metals and alloys — Stress corrosion testing — Part 1: General guidance on testing procedures*

ISO 7539-4, *Corrosion of metals and alloys — Stress corrosion testing — Part 4: Preparation and use of uniaxially loaded tension specimens*

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

ISO 3785, *Metallic materials — Designation of test specimen axes in relation to product texture*

ISO 6892-1, *Metallic materials — Tensile testing — Part 1: Method of test at room temperature*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 7539-1, ISO 8044, ISO 3785 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
test time
period between the start and the end of a test, with the criterion for the end being the initiation or failure of all test pieces, or the passage of an agreed test duration.

Note 1 to entry: The start of a test is when a specimen(s) is first exposed to the specified water chemistry, temperature and load.

4 Principle

The test consists of subjecting a specimen to uniaxial constant load in a specified and well-controlled environment for a set time period. The susceptibility to SCC is evaluated based on the time to initiation (for tests with in-situ monitoring), the time to failure, the extent of cracking (especially if no failure occurs) and/or the test time (when no cracking is observed). Because the test times can rarely be as long as the desired component lifetime, the test is carried out under accelerated test conditions of temperature, or test solution. The accelerating factor introduced shall not induce a change in crack initiation mechanism; the crack morphology shall be the same as expected or observed in the engineering application. The accelerating factor adopted should be appropriate for the intended application to evaluate the SCC susceptibility, but the accelerated nature of the laboratory initiation test can produce a different quantitative effect of materials, environments or stresses. For example, for a simulated boiling water reactor (BWR) water environment, the test may be accelerated by the addition of sodium sulfate (Na_2SO_4) or by the use of a crevice. On the other hand, for a simulated pressurized water reactor (PWR) primary water environment, the test may be accelerated by an increase in test temperature.

5 Specimens

5.1 General

5.1.1 General requirements

Shapes and dimensions of specimens shall be designed so as to ensure that SCC initiates in a gauge section or at a machined notch. Since SCC susceptibility is affected by microstructure and surface finishing, specimen orientation shall be categorized and deformation shall be minimised during specimen preparation.

5.1.2 Categories and shapes of specimens

The specimen type and geometry shall be based on existing test standards such as ISO 7539-4 or ISO 6892-1. Specimen types such as rod-shaped or plate-shaped tensile test specimens are recommended. Tubular or annular specimens may be tested, if appropriate.

5.1.3 Shapes and dimensions of grips/clevises

Grips/clevises shall match the linkage of the testing machine, which is often a threaded connection. For tensile specimens, load is often applied using pin or threaded connections.

When using pin loading, failure can occur at the pin hole, particularly in high strength materials, and the specimen shall be designed with this in mind. In general, the ligament surrounding the pin hole should be about twice the area of the gauge section, and the pin hole diameter should be at least as large as the thickest dimension of the gauge section.

5.1.4 Dimensions of specimen shoulder and radius

Failure can also occur by stress concentration in the radius transition from the gauge section to the grip section of tensile specimens, and a transition radius that is substantially larger than the gauge section is recommended. Undercut at the final transition to the gauge section shall also be avoided. The shapes in ISO 6892-1 provide excellent examples, but the underlying criterion is that failure should not occur in the radius nor predominantly in the adjacent gauge section. The following recommendations provide guidance for tensile loaded specimens.

a) For rod-shaped specimens, the radius of the shoulders (r) should be at least twice the diameter (d) of the parallel section.

b) For plate-shaped specimens, the radius of the shoulders (r) should be equal to or greater than the width of the parallel section (w).

5.1.5 Machined notch

Specimens with notches created mechanically in the parallel section may be used. The stress concentration and multiaxial stress create a higher stress than the nominal stress calculated using the minimum cross-sectional area of the bottom of the notch.

If the bottom of the notch is in the elastic region, the stress at the bottom of the notch should be evaluated by multiplying the nominal stress by the stress concentration factor K_t for the shape of the notch.

If the bottom of the notch is the plastic region, the stress distribution at the bottom of the notch changes due to plastic deformation, so it is desirable to evaluate the stress using elasto-plastic analysis such as finite element analysis.

5.1.6 Dimensions of gauge section

Dimension tolerances for preparing the gauge section of the specimen shall conform to ISO 6892-1. The diameter, thickness and width of the machined gauge section are usually constant, and shall be uniform over the entire length of the gauge section, specified in ISO 6892-1.

However, the gauge section of the specimen may be tapered towards the centre within tolerances specified in ISO 6892-1. By tapering the gauge section of the specimen, it is possible to increase the number of cracks at effective positions in the specimen's gauge section and to reduce breakages at the shoulder sections of the specimen.

Specimens having tapered gauge lengths may be employed for the purpose of obtaining a range of initial stresses.

When a specimen shape other than those specified in ISO 6892-1 is necessary, the specimen shape should ideally have a gauge length of 10 mm or more and a width of 3 mm or more in the parallel section.

With agreement among the parties involved in the testing, specimens that are smaller than those described above may be used. Without careful consideration, it is possible that the results of the SCC test can be greatly influenced by the cross-sectional area of the specimen and the area exposed to the environment.

Caution has to be exercised in the case of miniature specimens, as machining becomes difficult when the cross-sectional area of the specimen decreases, and elevated susceptibility to stress concentration can result from pitting, general corrosion, bending, etc. The net section stress also rises rapidly as cracks form. When using miniature specimens, it is recommended that the diameter and thickness of the gauge section of the specimen be at least 6 times the grain size.

In general, larger specimens are preferred in terms of the reproducibility and relevance of the test results.

5.1.7 Serial loading of multiple specimens

The constant load test may be performed by connecting multiple specimens in series. In this case, a load-catch jig designed to prevent unloading after fracture of one specimen shall be used, or the time of unloading be detected. Load-catch mechanisms need to consider whether shock re-loading to the remaining specimens can occur after the failure of one specimen.

5.2 Preparation of specimens

5.2.1 Orientation of specimen sampling

The orientation of the specimen should account for the orientation of cracks of concern in the application of interest. Often, specimens are machined so that the longitudinal direction of specimens matches the rolling direction. The sampling direction and location of the specimens should be determined by agreement among the parties involved in the testing.

The test specimen axes in relation to product texture should be designated by an X-Y-Z orthogonal coordinate system specified in ISO 3785. The letter X denotes the direction of principal deformation (maximum grain flow in the product). The letter Y denotes the direction of least deformation. The letter Z denotes the direction normal to the X-Y plane. The anticipated direction of crack extension for notched specimens should be designated using a hyphenated code wherein the letter(s) preceding the hyphen represent the longitudinal direction of the specimens and the letter(s) following the hyphen represent the anticipated direction of crack extension. For unnotched specimens, surfaces normal to the anticipated direction of crack development are specified in plate-shaped specimens and are not specified in rod-shaped specimens. The specified surface in plate-shaped specimens should be designated using a hyphenated code wherein the letter(s) preceding the hyphen represent the longitudinal direction of the specimens and the letter(s) following the hyphen represent the direction normal to the surface.

The orientation of specimens sampled from test products is designed according to ISO 3785. The orientations of unnotched plate-shaped specimens for sheet, plate, rectangular bar, cylinder and tube, are shown in Figure 1. When the specimen direction is aligned with the product's characteristic grain-flow directions, a single letter for each case is used to denote the direction perpendicular to the crack plane and the direction of intended crack extension, as shown in Figures 1 a), b) and c). When the specimen orientation directions lie midway between the product's characteristic grain-flow directions, two letters shall be used to denote the normal to the crack plane or the crack propagation direction, as shown in Figure 1 d).

Designations by an L-T-S orthogonal coordinate system for rectangular sections and by an L-R-C orthogonal coordinate system for cylindrical sections specified in ASTM E399-20a[1] are described together in Figure 1. The designations of specimen sampling orientations are compared to ISO 3785, ASTM E399-20a[1] and ASTM A370-20[2] in Annex A.

NOTE For rectangular sections, the definition of orientations in ASTM E399-20a[1] and ASTM A370-20[2] corresponds to that in ISO. For cylindrical sections, the definition of orientations in ASTM does not always correspond to that in ISO because that in the ASTM standards is a geometry-based system.

When there is no grain-flow direction as in a casting, specimen location and crack plane orientation shall be defined on a part drawing and the test result shall carry no orientation designation.

For plate-shaped specimens, the most relevant orientation is generally when the test specimen is parallel to the rolling surface of the plate.

In the case of a use of cold or warm rolling to accelerate tests for material comparisons, the orientation of the specimen for the rolling direction also should account for the orientation of cracks.

5.2.2 Methods of specimen sampling

Specimens can be extracted by sawing, electrical discharge machining, cutting, or grinding, provided that damage to the material is minimised and constrained to the surface.

5.2.3 Surface finishing

Significant effects of surface conditions on the SCC initiation time are well known, and thus surface finish needs to be carefully considered and well controlled. Concerns include physical defects, near-surface microstructural changes (including plastic deformation and possible formation of nanocrystalline layer), residual stress, increased hardness, and chemical contamination.

The increase of stress due to surface asperity usually has more of an effect on the SCC of high strength materials than low strength materials.

Unless it is necessary to evaluate the as-supplied or as-fabricated surface, the material should be tested with a reproducible surface finish by mechanical grinding or polishing, electropolishing or chemical polishing.

Prior to testing the specimen shall be degreased using a solvent such as acetone and washing using ethanol and high-purity water.

5.2.4 Mechanical grinding and polishing

Unless a manufacturing surface is being evaluated, the surface of a specimen should be carefully prepared and controlled. A common surface preparation is the sequential use of coarser-to-finer abrasives, and using cooling fluid is preferred, as described in ISO 3366 and ISO 21948. The final finish, grinding or polishing should be agreed between the parties but as a default, a final finish of P600 is recommended. The final grinding or polishing should be undertaken preferably in the longitudinal direction of the specimen. However, other grinding and polishing conditions may be adopted by agreement between the parties involved in testing.

In final machining and surface preparation, aggressive material removal and overheating should be avoided to minimise residual stress and changes of microstructure on the surface.

Care should also be taken to minimise surface contamination from polishing residue.

5.2.5 Electrolytic or chemical polishing

Electrolytic or chemical polishing may be appropriate as they can reduce the machined layer or surface roughness that result from the mechanical finishing of the surface. It should not be assumed that electropolishing or chemical polishing always removes surface damage and defects, or that pitting or localized corrosion does not occur during the process.

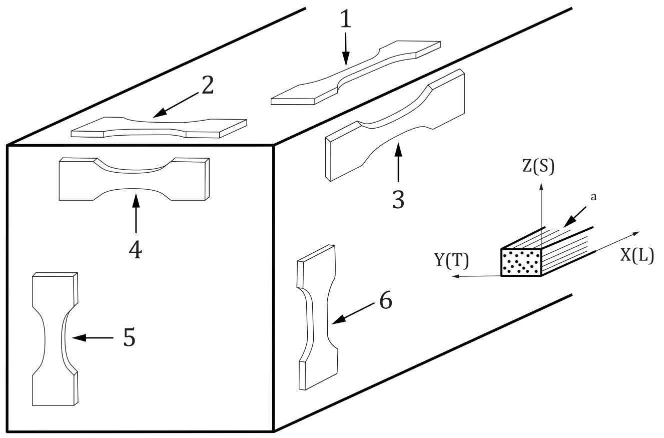
If the final surface finish is done using chemical treatment (polishing, etching, etc.), attention should be taken when selecting conditions to avoid selective dissolution and the formation of undesirable residue on the surface.

Chemical or electrochemical processes that generate hydrogen should not be used on materials that are sensitive to hydrogen induced damage.

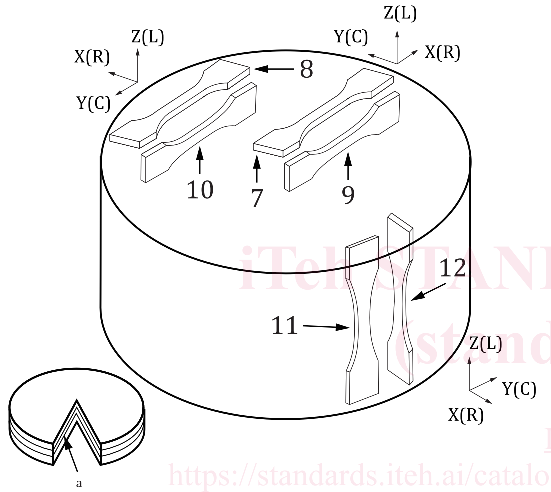
5.2.6 Marking

If it is necessary to mark specimens for identification, marks shall be put as far away as possible from the areas where SCC initiation occurs and at positions that do not affect the test results.

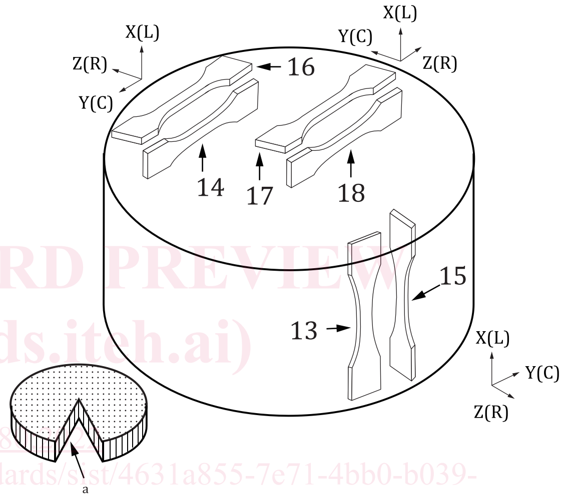
Ends of an original gauge section may be marked with a fine mark or scribed line. Note that marking by a punch shall be avoided because it can lead to premature fracture.



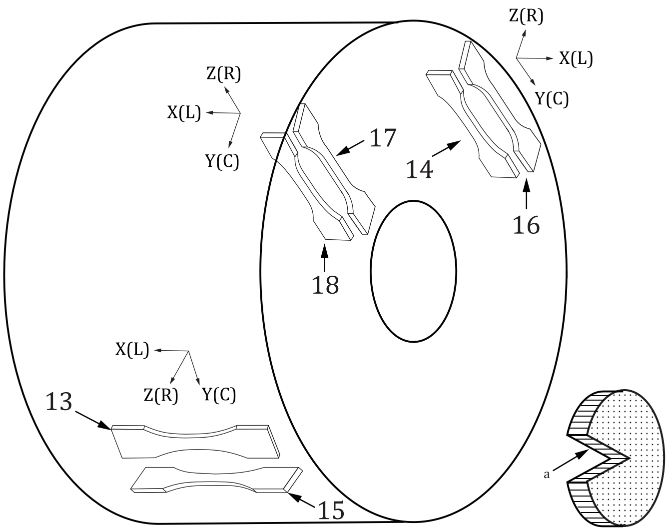
a) Sheet, plate, rectangular bar



b) Cylinder — Radial grain flow



c) Cylinder — Axial grain flow



d) Tube (axial grain flow)