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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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Contents

Page

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

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.

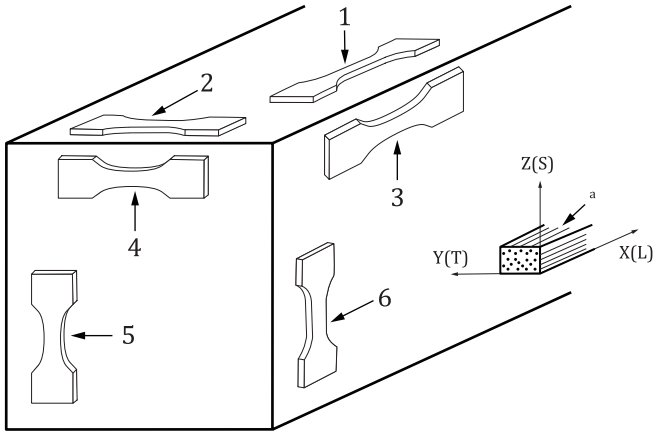
If the final surface finish is done using chemical treatment (polishing, etching, etc.), attention should be taken when selecting conditions to avoid selective dissolution and the formation of undesirable residue on the surface.

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

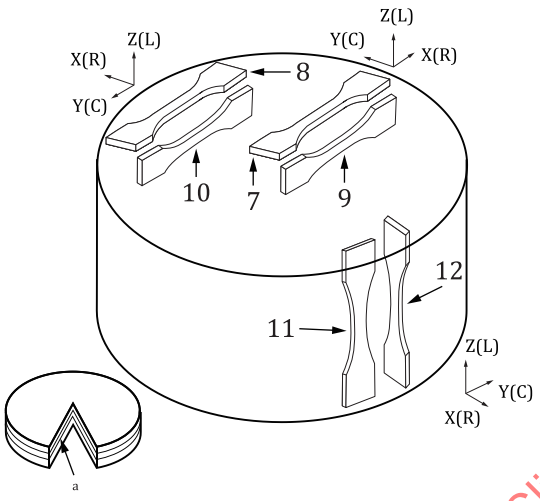
5.2.6 Marking

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.

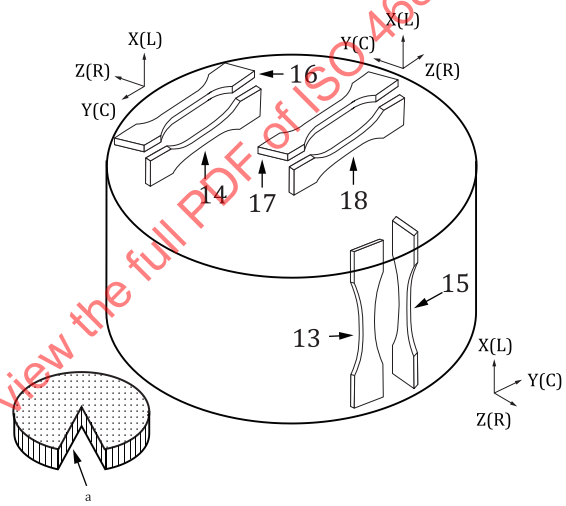
Ends of an original gauge section may be marked with a fine mark or scribed line. Note that marking by a punch shall be avoided because it can lead to premature fracture.



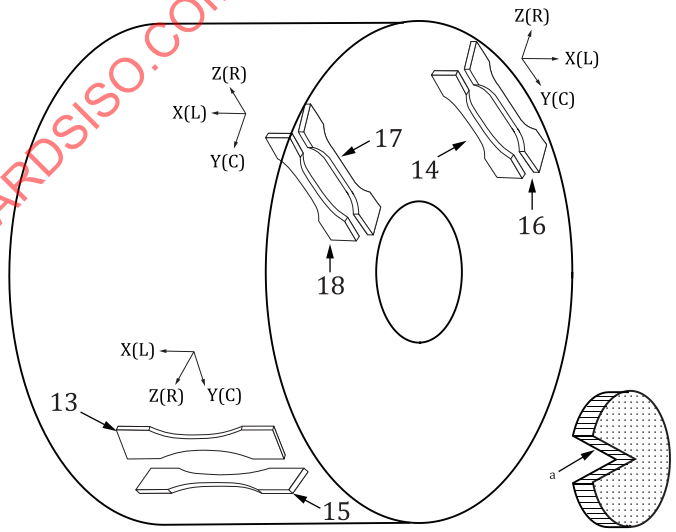
a) Sheet, plate, rectangular bar



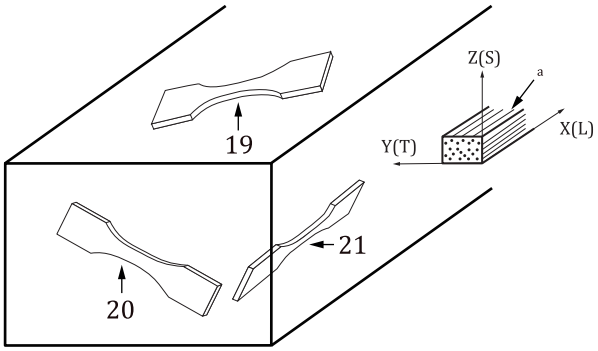
b) Cylinder — Radial grain flow



c) Cylinder — Axial grain flow



d) Tube (axial grain flow)



e) Not aligned

Key

1	X-Z (L-S) orientation specimen	13	X-Z (L-R) orientation specimen
2	Y-Z (T-S) orientation specimen	14	Y-Z (C-R) orientation specimen
3	X-Y (L-T) orientation specimen	15	X-Y (L-C) orientation specimen
4	Y-X (T-L) orientation specimen	16	Y-X (C-L) orientation specimen
5	Z-X (S-L) orientation specimen	17	Z-X (R-L) orientation specimen
6	Z-Y (S-T) orientation specimen	18	Z-Y (R-C) orientation specimen
7	X-Z (R-L) orientation specimen	19	XY-Z (LT-S) orientation specimen
8	Y-Z (C-L) orientation specimen	20	YZ-X (TS-L) orientation specimen
9	X-Y (R-C) orientation specimen	21	X-YZ (L-TS) orientation specimen
10	Y-X (C-R) orientation specimen	*X*	direction of principal deformation (maximum grain flow in the product)
11	Z-X (L-R) orientation specimen	*Y*	direction of least deformation
12	Z-Y (L-C) orientation specimen	*Z*	direction normal to the X-Y plane
a	Grain flow.		

Figure 1 — Convention for recoding orientation of unnotched plate-shaped specimens

6 Test apparatus

6.1 Test set-up

A typical test configuration is shown in Figure 2. It consists of a device that applies load to a specimen, a test chamber for the specimen and the environment, and a circulation system including a water quality control and monitoring. The test chamber is an autoclave designed for the high-temperature and high-pressure environments that simulate the light water reactor water environments. The water control and monitoring system is typically at normal temperature and pressure. The wetted system materials are typically austenitic stainless steel or materials having higher corrosion resistance.

6.2 Loading mechanism

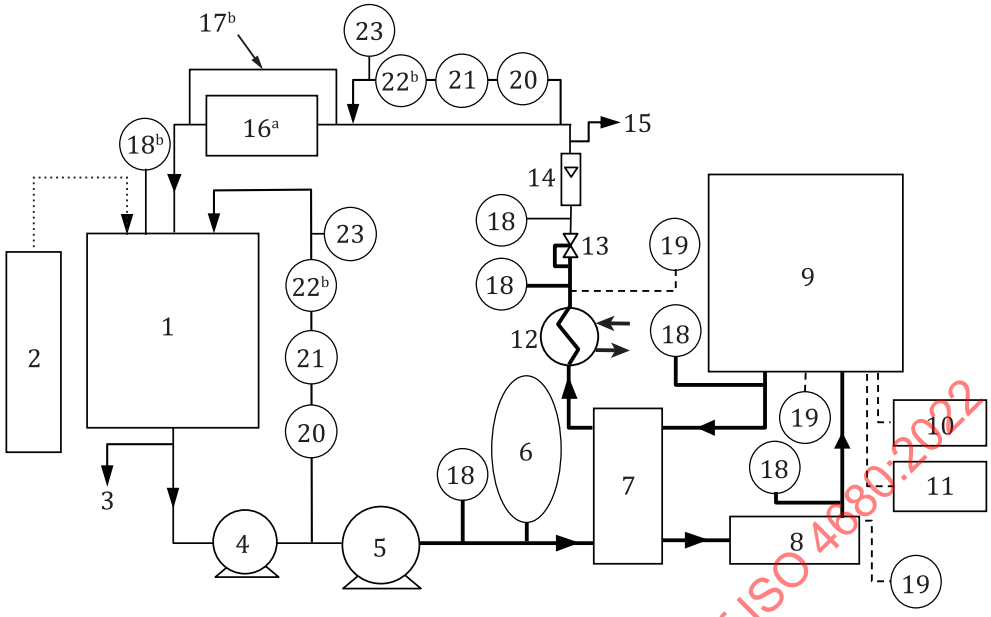
The loading device can be a mechanical loading system, a spring, a lever (cantilever), internal pressure loading, or an air cylinder. A device that can load uniaxial tensile load with at least a ±1 % accuracy is used. The force applied shall have passed the tolerance specified in ISO 7500-1 or ASTM E4.[6] SCC initiation can be sensitive to small decreases in load during testing, and thus highly controlled, active loading is preferred.

6.3 Autoclave

The autoclave shall be a container capable of sealing and holding the target high-purity water at high temperatures. The material of the autoclave shall be austenitic stainless steel or other corrosion resistant material. A heater and temperature controller shall be used to control the temperature inside the autoclave. The difference in temperature in the autoclave where specimens are located should be within ±3 °C of the target value. For safe use of the autoclave, refer to the performance requirements specified in ISO 16528-1 and ISO 16528-2.

6.4 Circulating loop

The water circulation loop is equipped with a high-pressure pump that feeds the controlled test solution to the autoclave, and has a pressure regulating valve that can maintain the desired pressure. To measure the water quality (see Annex B), install measuring devices on the inlet and outlet sides of the autoclave as shown in Figure 2. The inlet side is measured at the normal temperature and low-pressure section from the water-chemistry-controlling reservoir to the high-pressure pump. The autoclave outlet side is the normal temperature and low-pressure part.



Key

1	water-chemistry-controlling reservoir	13	pressure-regulation valve
2	regulating gas injection system	14	flow meter
3	inlet water sampling point	15	outlet water sampling point
4	feed water pump	16	ion exchange resin
5	high-pressure pump	17	bypass line
6	accumulator	18	pressure meter
7	heat exchanger	19	thermocouple
8	preheater	20	solution conductivity meter
9	test chamber (autoclave with loading mechanism)	21	dissolved oxygen analyser
10	crack-initiation-detecting device	22	dissolved hydrogen analyser
11	corrosion-potential-measuring device	23	pH meter
12	cooler		

Continuous line water line
Bold line high-pressure section of water line
Dashed line electrical instrumentation
Dotted line gas line

[a] Unessential in the simulated PWR primary water system.
[b] Unessential in the simulated BWR water system.

Figure 2 — Schematic of UCL test equipment configuration for evaluating SCC in simulated light water reactor water environment

7 Experimental procedure

7.1 Test environment

7.1.1 General

By adjusting the water chemistry of the water-chemistry-controlling reservoir and circulating the test water through the autoclave containing the specimen, the water chemistries at the inlet and outlet of the autoclave are set as the target test conditions and should be as identical as possible and appropriate

to the purposes of the test. The simulated BWR water environments often represent normal water chemistry (NWC) or hydrogen water chemistry (HWC). Examples of the water chemistry of the simulated BWR water environment and PWR primary water environment test under the accelerated condition are shown in Annex C. For tests in the simulated BWR water environments (see Table C.1), the autoclave outlet water should flow through the ion-exchange resin to eliminate impurity ions. Then, the test water is returned to the water-chemistry-controlling reservoir. In simulated PWR primary water environments, if the test water contains borate ions and lithium ions (see Table C.2), the autoclave outlet can directly return to the reservoir, or (preferably) flow through an ion-exchange resin that is equilibrated to the desired water-chemistry.

7.1.2 Corrosion potential

Measurement of the corrosion potential of the specimen or a separate electrode versus a reference electrode should employ a high-input-impedance electrometer. The electrodes shall be electrically isolated. The measurement of the corrosion potential should be performed throughout the test.

7.1.3 Measurement parameters

The parameters shown in Annex B should be measured during the test. There are differences in some parameters for the simulated BWR water environment test and the simulated PWR primary water environment test due to the difference in the water chemistries.

7.2 Specimen number and applied stress

The number of test specimens per condition shall be multiple as determined by agreement between the parties according to the test purpose. When comparing the SCC susceptibility of different materials using the time to failure, it is desirable from a statistical perspective that the number of test specimens should be seven or more.

The initial stress applied to the specimen should be determined by dividing the applied load by the initial cross-sectional area of the gauge section in the specimen. If the dimensions of the gauge section are within the tolerances defined in ISO 6892-1, the nominal dimension may be used for calculating the original cross-sectional area. If they are not within the tolerances, the dimensions of all specimens shall be measured.

For a specimen having a tapered gauge length, the range of initial stresses applied to the specimen should be agreed between the parties. The minimum and maximum initial stresses are determined by dividing the applied load by the maximum and minimum initial cross-sectional areas of the gauge section in the specimen, respectively. The dimensions of all specimens shall be measured.

7.3 Testing procedures

7.3.1 General

The testing procedure is as followed. The specimen is installed and set in the autoclave, and water chemistry is adjusted in the autoclave with the circulation system and the desire pressure achieved. The temperature in the autoclave shall then be increased and controlled at the test temperature before applying load. If a specimen with a crevice is used, the crevice is created before installation. The test is terminated when the number of specimens that have failed reaches an agreed threshold. In the case of monitoring using an electrical resistance measurement (see 7.5), the test is terminated when the number of specimens for which the time to initiation has been detected by the monitoring reaches an agreed threshold. Alternatively, the test may be terminated once the duration passes an agreed threshold. The threshold for the number of specimens and the test duration should be pre-determined by the interested parties. After the test duration passes, the heater is shut down to decrease temperature. If the specimen has not failed, it should be unloaded before the heater is shut down to avoid an overload of the specimen from thermal shrinkage difference with pull rods should the systems trip. When the temperature decreases to room temperature, the pressure in the system is decreased to normal pressure, and the specimen is removed. The procedure of loading and unloading is determined

on the basis of discussion between interested parties for the test objectives. Ideally, the test is not interrupted until all specimens fail. The test may be interrupted on the basis of discussion between interested parties and the consideration of the effect on the test results. Recommended procedures for the interruption and restart are detailed in Annex D.

7.3.2 Test solution adjustment

After installation of the specimens, water chemistry including dissolved oxygen concentration and dissolved hydrogen concentration shall be adjusted to the specified chemistry in the circulation system.

7.3.3 Temperature control

After the desired pressure in the system is achieved, the water in the autoclave is heated. The temperature ramp rate is recommended to be at least 50 °C/hour to limit corrosion during heat up. Overshooting is undesirable, so often it is wise to heat to a temperature 10 °C to 20 °C below the test temperature, then increase it. To prevent boiling or the creation of a steam bubble near the top of the autoclave, the system pressure should provide a margin of at least 10 °C. Unintended stressing of the specimens by the difference in the thermal expansion coefficient to the loading equipment or test jigs should be considered.

7.3.4 Loading

If the specimen is loaded by an external loading system, this is done after stable water temperature and chemistry is achieved in the autoclave. External loading systems can be a servo system, spring system, lever system, internal pressure system, or air cylinder system. Note that some internal pressure systems loaded by the difference in pressure inside and outside the autoclave should consider making the final increase in system pressure after stable conditions are achieved. Seal resistance for pull-rods should also be accounted for.

7.3.5 Unloading

At the end of the pre-determined test duration, the specimen should be unloaded, and subsequently the temperature decreased.

7.3.6 Completion of test

When the pre-determined number of specimens have failed, when the time to initiation has been detected on the pre-determined number of specimens, or when the pre-determined test duration is achieved, the test temperature is decreased to room temperature and the pressure is decreased to ambient pressure. After that, the circulation system is stopped, the autoclave is drained and opened, and the specimens are removed. Specimens should be cleaned using high-purity water or ethanol and then dried. Characterisation of the specimen can now be performed to confirm if SCC has occurred.

7.4 Addition of crevice

7.4.1 General

The crevice design is based on agreement between the interested parties. A crevice facilitates the formation of a favourable environment for SCC to initiate, and has the largest effect in oxidizing environments.

7.4.2 Materials of crevice formation

Crevice forming materials shall be stable in the test environment and shall create a geometry that strongly limits mass transfer in a repeatable manner. As a crevice forming material, unwoven fabric or metal foil is recommended for use. For example, high-purity-graphite-fibre unwoven fabric or stainless steel foil is used. The metallic crevice forming material should be of the same metal as the specimen.

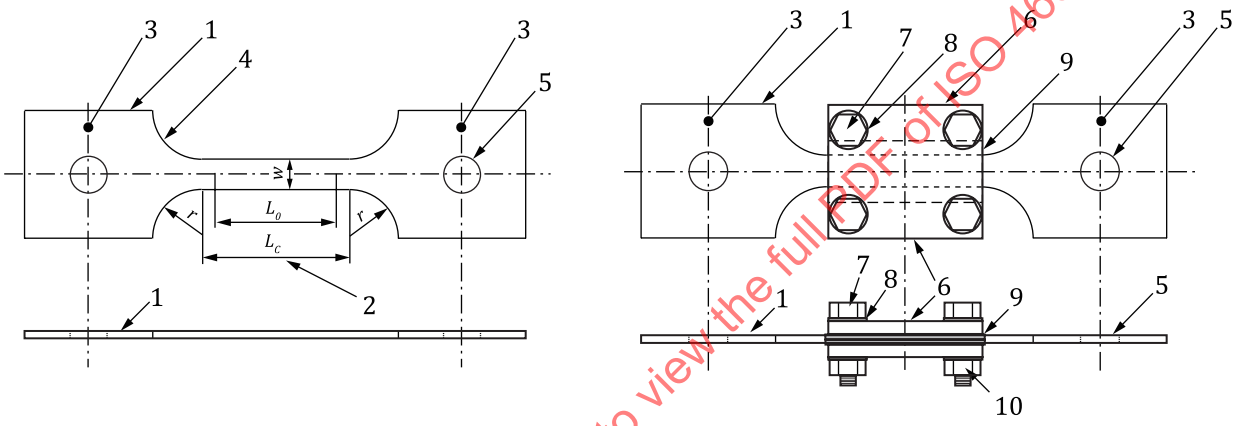
Impurities associated with the crevice-forming material can impact the crack initiation process. Appropriate procedures, such as prior rinsing or immersion in high-purity water, should be undertaken to remove these impurities.

7.4.3 Procedure of crevice addition

The specimen and the crevice forming material, including any fixtures, shall be degreased using acetone, ethanol, and high-purity water. The crevice forming material is attached on the specimen with the fixtures by bolts and nuts, as shown in Figure 3.

If metal foil is used as the crevice forming material, the metal foil should be tightly wrapped around the parallel section of the specimen.

If unwoven fabric is used as the crevice forming material, the crevice height should be set to provide an apparent density of pressed fabric, as determined by dividing weight of the fabric per unit area by the crevice height, to ensure a robust crevice.



a) Specimen **b) Specimen with crevice**

Key

1	specimen	8	washer
2	parallel section	9	crevice forming material
3	grip section	10	nut
4	shoulder portion	w	width of parallel section
5	pin hole	r	radius of shoulder portion
6	fixture	L_0	original gauge length
7	bolt	L_c	parallel section

Figure 3 — Example of the specimen with crevice by carbon fabric and fixtures

7.5 Electrical resistance measurement methods

7.5.1 General

The general use of direct current (DC) and alternating current (AC) potential drop methods for crack monitoring and possible sources of error are described in ISO 11782-2 and ISO 21153, with supplementary information included in Annex E. The crack initiation time is judged by monitoring the potential drop during the test.

7.5.2 Measurement requirements

While either DC potential drop (DCPD) or AC potential drop (ACPD) may be used, much greater care needs to be taken with the AC method to avoid perturbation of leads and extraneous sources of noise. The use of reversing DCPD reduces noise and removes thermoelectric effects, and modern "integrating" instruments give very stable measurements. For ACPD, the critical issue is to reduce AC pick up (voltages induced in the signal) from the current supply leads. In the case of the DCPD technique, the potential drop reflects the cross-sectional area of the defect. As the AC frequency is increased, the current flows through a thinner layer near the surface of the specimen (skin effect) and therefore "sees" a smaller effective cross-section. However, reversing DCPD provides dramatically higher noise rejection and sophisticated implementations of both have shown DCPD to be superior and dramatically less challenging and expensive to implement. Also, for probes placed near a defect or notch, the advantage of the ACPD skin effect is minimal.

The positioning of probes attached to a specimen also affect the sensitivity. The closer the probes are placed to the location of cracking, the greater the sensitivity.

8 Assessment of results

8.1 Time to failure

The most commonly used evaluation index for susceptibility of SCC is time to failure. This can be determined on the basis of the load and displacement variation of the test apparatus accompanying the failure of the specimen, or other crack detection techniques (such as DC potential drop).

8.2 Validity of failure position

For the failure position of the specimen, the failure at a position in the original gauge section of the specimen is treated as valid, while the failure at the outside of the original gauge section is treated as invalid. If the failure positioned near the end of the gauge section, there should be some concern. The original gauge section is specified in ISO 6892-1. When a specimen shape other than those specified in ISO 6892-1 is necessary, an original gauge section should be decided as a section without strain derived from the machined radius.

8.3 Other evaluation indexes

If no fracture occurs, cracks can occur in the gauge section of the specimen, even if propagation does not appear to progress. For specimens that do not experience full fracture, cracks should be investigated by visual examination or scanning electron microscopy, or by cross sectional metallography. The methodology and magnification for different test materials should be determined by agreement among the parties involved in the testing. In addition, when observing the surface of the specimen, corrosion products may be removed if necessary (refer to ISO 8407).

The number of cracks per unit length of the specimen may be used for comparative evaluation of SCC susceptibility. When using such a method, metallurgical examination (e.g. with a stereo optical microscope, digital microscope, or scanning electron microscope (SEM)) on the specimen surface or cross section can detect small cracks that cannot be seen with the naked eye. It is desirable to apply such examination along with the method to determine the number of cracks.

When the specimen fails, the maximum SCC depth may be measured on the fractured surface. Specimens that did not fracture may be measured after post-test fracture in tension or by fatigue loading.

8.4 Observation of crack morphology

The crack morphology should be determined at a distinguishable magnification by stereo optical microscope and/or SEM to differentiate intergranular cracking, transgranular cracking, and dimple-ductile cracking. Other cracking parameters, such as the area of the fracture surface and the maximum crack length, should also be recorded.

8.5 Detection of crack initiation using electrical resistance measurement methods

Crack initiation can be detected using electrical resistance measurement methods (see 7.5 and Annex E). The measurement systems for crack initiation are required to achieve high resolution potential drop monitoring by eliminating noise and extensive data averaging. The crack initiation time is judged from monitoring results throughout the test. Defining crack initiation from potential drop data has some uncertainty because the potential can change slowly, although it is always more precise than other techniques.

9 Test report

The test report shall include the following information:

a) a full description of the test material, including type of materials, chemical composition, heat treatment, and microstructural condition;

b) position, orientation, and methods of specimen sampling;

c) shape, dimension, surface finishing of specimens, and notch parameters such as depth and root radius if a machined notch is applied;

d) position of a weld metal if a weld joint is used and reduction of cold working if a cold-worked material is used;

e) the International Standard used (including its year of publication);

f) number of specimens;

g) procedures of loading and temperature controlling;

h) applied load and applied stress;

i) test temperature and test pressure;

j) measured values of test solution described in Annex B;

k) procedure of crevice addition and materials of crevice formation;

l) time to failure and test time, or initiation time in the case of measuring the crack initiation time;

m) judging methods of failure, positions of failure in specimens, morphology of cracking, and number of unfailed specimens;

n) any deviations from the procedure;

o) any unusual features observed;

p) the starting and ending date of the test;

q) special notes.

Optional reported items can be decided by reference to Annex F.

Annex A
(informative)

A comparison of specimen orientations in standards

Definitions of orientations for specimen sampling that differ between ISO and ASTM standards are summarised. The orientations for specimen sampling from the various materials specified in this document are addressed by comparison with those in other standards.

A.1 Definition of orientations

A.1.1 X-Y-Z orthogonal coordinate system specified in ISO 3785

For rectangular and cylindrical sections, the system is used for rolled plate, sheet, extrusions and forgings with non-symmetrical grain flow.

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.

A.1.2 L-T-S orthogonal coordinate system specified in ASTM E399-20a[1]

For rectangular sections, the system is used for rolled plate, sheet, extrusions, and forgings with non-symmetrical grain flow.

The letter L denotes the direction of principal deformation (maximum grain flow in the product).

The letter T denotes the direction of least deformation.

The letter S denotes the direction normal to the L-T plane.

A.1.3 L-R-C orthogonal coordinate system specified in ASTM E399-20a[1]

For cylindrical sections, the system is useful for extruded or forged parts having circular cross section, regardless of the maximum grain flow depending on the manufacturing method.

The letter L denotes the axial direction.

The letter R denotes the radial direction.

The letter C denotes the circumferential or tangential direction.

A.2 Orientations for specimen sampling

The orientations for specimen sampling from various materials described in ISO and ASTM standards are compared in Table A.1. The orientations defined in this document are consistent with those in ISO 3785. The orientations of unnotched plate-shaped specimens are additionally defined in this document. The orientations defined in ASTM A370-20[2] and ASTM E399-20a[1] are for informational purposes only.

Table A.1 — Orientations for specimen sampling from various materials described in ISO and ASTM standards

Specimen type	Specimen direction for grain flow	Shape and grain flow direction	Standard			
			ISO 4680	ISO 3785	ASTM A370-20	ASTM E399-20a
Unnotched specimens (Rod-shaped)	Aligned	Sheet, plate, rectangular bar	Referred from ISO 3785	Figure 1 a)	Figure 1[a]	—
		Cylinder, Radial grain flow	Referred from ISO 3785	Figure 1 b)	Figure 2 c) d) [b]	—
		Cylinder, Axial grain flow	Referred from ISO 3785	Figure 1 c)	Figure 2 a) [c] Figure 2 c) d) [b]	—
		Thick-walled tubes	Referred from ISO 3785	Figure 1 d)	Figure 2 b) [d]	—
		Thin-walled tubes, helical grain flow	Referred from ISO 3785	Figure 1 e)	—	—
	Not aligned	Sheet, plate, rectangular bar	Referred from ISO 3785	Figure 1 f)	—	—
	No grain flow	Casting	Referred from ISO 3785	Defined in a part drawing	—	—
Unnotched specimens (Plate-shaped)	Aligned	Sheet, plate, rectangular bar	Figure 1 (a)	—	Figure 1[a]	—
		Cylinder, Radial grain flow	Figure 1 (c)	—	—	—
		Cylinder, Axial grain flow	Figure 1 (d)	—	—	—
		Thick-walled tubes	Figure 1 (b)	—	—	—
	Not aligned	Sheet, plate, rectangular bar	Figure 1 (d)	—	—	—
	No grain flow	Casting	Defined in a part drawing	—	—	—
Notched specimens	Aligned	Sheet, plate, bar	Referred from ISO 3785	Figure 2 a)	Figure 1[a]	Figure 1 a)
		Cylinder, Radial grain flow	Referred from ISO 3785	Figure 2 b)	—	Figure 1 c) [e]
		Cylinder, Axial grain flow	Referred from ISO 3785	Figure 2 c)	—	Figure 1 c) [e]
	Not aligned	Sheet, plate, bar	Referred from ISO 3785	Figure 2 b)	—	Figure 1 b)
	No grain flow	Casting	Referred from ISO 3785	Defined in a part drawing	—	Defined in a part drawing

[a] ASTM A370: Longitudinal and transverse test specimens from general wrought products.

[b] ASTM A370: Tangential test specimens from disc and ring forgings.

[c] ASTM A370: Longitudinal, radial and tangential test specimens from shafts and rotors.

[d] ASTM A370: Longitudinal and tangential test specimens from hollow forgings.

[e] ASTM E399: Specimen location and crack plane orientation shall reference original cylindrical section geometry such that the L direction is always the axial direction for the L-R-C system, regardless of the maximum grain flow.

Annex B
(normative)

Measuring items of test solution

The water chemistry parameters in Table B.1 should be measured during the test at a suitable timing. The timing of measurement for each item is classified into category A, B, or C. There are differences in some of the measured items for the simulated BWR water environment test and the simulated PWR primary water environment test due to differences in the water chemistry for the tests.

Table B.1 — Measured parameters of test solution

Measured parameters	Simulated BWR water condition	Simulated PWR primary water condition
Conductivity	Category A [a]	Category B [c]
Dissolved oxygen concentration (inlet)	Category A [c]	Category B [c]
Dissolved oxygen concentration (outlet)	Category A [a]	Measured as necessary [a]
Dissolved hydrogen concentration (inlet)	Measured as necessary [c]	Category B [c]
Dissolved hydrogen concentration (outlet)	Measured as necessary [a]	Measured as necessary [a]
Specified temperature	Category A	Category A
Corrosion potential	Measured as necessary [d]	Measured as necessary [d]
Borate ion concentration	—	Measured as necessary [b]
Lithium ion concentration	—	Measured as necessary [b]
Sulfate ion (SO_4^{2-}) concentration	Category C [b]	Measured as necessary [b]
Chloride ion (Cl^-) concentration	Category C [b]	Measured as necessary [b]
Fluoride ion (F^-) concentration	Measured as necessary [b]	Measured as necessary [b]
pH	Measured as necessary [a]	Measured as necessary [b]

Category A refers to continuously measured parameters.

Category B refers to items measured at the beginning of the test.

Category C refers to items measured one or more times during the test.

[a] Measured at outlet position (at ambient temperatures and under ordinary pressure) near autoclave.

[b] Sample test solution at outlet position (at ambient temperatures and under ordinary pressure) near autoclave.

[c] Measured for test solution in reservoir or tank.

[d] Measured for a specimen or a separate electrode in test chamber, and reported the reference electrode used.

Annex C
(informative)

Water chemistry conditions and measurement items for simulated BWR and PWR primary water environment tests

The simulated BWR water environment may exhibit normal water chemistry (NWC) or hydrogen water chemistry (HWC). An example of the water chemistry for the simulated BWR water environment test under the NWC condition is shown in Table C.1. Examples of the water chemistry for the simulated PWR primary water environment test are shown in Table C.2. Temperatures A, B and C for the PWR test indicate the temperature at the outlet of the reactor pressure vessel, which at the outlet of the steam generator and the typical temperature acceleration condition, respectively. Environment I provides the initial water chemistry condition in consideration of the extension of the running cycle. Environment II provides the typical average water chemistry during the operation.

Table C.1 — Example of water chemistry for simulated BWR water environment test (sulfate ion accelerated NWC)

Analysis parameters	Controlled values for water chemistry
Temperature	288 °C
Conductivity (outlet) [a]	(0,10 ± 0,01) mS/m (at 25 °C)
Dissolved oxygen concentration	(8,0 ± 1,0) mg/l
Chloride ion (Cl^-) concentration	≤5 µg/l
Sulfate ion (SO_4^{2-}) concentration [b]	Added as Na_2SO_4 to control the outlet conductivity
Test pressure should be set above 8 MPa to avoid boiling of test solution.	
[a] High-purity water as raw water should have conductivity less than 0,007 mS/m (at 25 °C).	
[b] Electrical conductivity at outlet position near autoclave should be continuously controlled through addition of sodium sulfate (Na_2SO_4).	

Table C.2 — Examples of water chemistry for simulated PWR primary water environment test

Analysis parameters	Controlled values for water chemistry	
Temperature	Temperature A	(325 ± 3) °C
	Temperature B	(290 ± 3) °C
	Temperature C	(360 ± 3) °C
Conductivity	Environment I	(3,5 ± 0,3) mS/m (at 25 °C)
	Environment II	(2,15 ± 0,3) mS/m (at 25 °C)
pH	Environment I	6,25 ± 0,15 (at 25 °C)
	Environment II	7 ± 0,15 (at 25 °C)
Borate ion concentration (as H_3BO_3 is added)	Environment I	(1 800 ± 180) mg/l (as B)
	Environment II	(500 ± 50) mg/l (as B)
Lithium ion concentration (as LiOH is added)	Environment I	(3,5 ± 0,35) mg/l
	Environment II	(2 ± 0,2) mg/l
Dissolved oxygen concentration	≤5 µg/l	
Dissolved hydrogen concentration	(30 ± 5) cm^3/kg [a]	

Table C.2 *(continued)*

Analysis parameters	Controlled values for water chemistry
Sulfate ion (SO_4^{2-}) concentration	≤50 µg/l
Fluoride ion (F^-) concentration	≤50 µg/l
Chloride ion (Cl^-) concentration	≤50 µg/l
[a] Under STP (0 °C, 101,325 kPa).	

Annex D
(informative)

Procedures to interrupt and resume testing

D.1 Effect of interruption on SCC behaviour

Variation of loading and environment by interruption can affect SCC behaviour. Repetition of unloading and reloading possibly shortens initiation time in general.

D.2 Procedures to interrupt and resume testing

D.2.1 General

Test interruption is broken down in the following table. The advisable procedure is summarised in Table D.1.

Table D.1 — Categories and desirable procedures of test interruptions

Category	Objective	Desirable procedure
Planned interruption	Inspection of instruments (except autoclave)	Remove the applied load, and then decrease the temperature and pressure. Keep specimens in the test solution; do not remove them.
	Opening and inspections of autoclave	Remove the applied load, then decrease the temperature and pressure, and finally remove specimens. In the case of creviced specimens, keep them in high purity water.
Unplanned interruption	—	Confirm the load on the specimens during the interruption. Keep specimens without an overload in the test solution if they do not need to be removed or eliminate overloaded specimens from the evaluation.

D.2.2 Procedures to interrupt testing for planned interruption

The specimen shall be unloaded to avoid overloading in the interruption process. The temperature shall be decreased before pressure is decreased. If uninstallation is needed, specimen should be taken out of the autoclave after releasing water and the opening the autoclave. If a specimen with a crevice is used, it should be stored in room temperature high-purity water.

D.2.3 Procedures to interrupt testing for unplanned interruption

In the case of unplanned interruption, the specimen can be overloaded. The test results for specimens with overloading shall be eliminated. The structure and equipment to avoid overloading are recommended.

D.2.4 Procedures to resume testing

If a specimen is uninstalled, it is installed again in the autoclave. The water is circulated and water chemistry including dissolved oxygen concentration and dissolved hydrogen concentration is adjusted to the test condition. The pressure in the circulation system and autoclave is adjusted by a high-pressure pump and subsequently temperature is increased. Over-heating and overloading shall be avoided.

If the specimen is loaded by an external loading system, it shall be loaded after confirmation of the stable water temperature in the autoclave.

D.3 Report

If a test was interrupted and then resumed, the following shall be reported as a special note.

a) Elapsed time from start to interruption of testing;

b) procedure of test interruption;

c) storing of specimens until resuming testing;

d) procedure of resuming testing.