



BSI Standards Publication

**Corrosion of metals and alloys — Test method
for measuring the stress corrosion crack
growth rate of steels and alloys under static-
load conditions in high-temperature water**

**Corrosion of metals and alloys —
Test method for measuring the
stress corrosion crack growth rate
of steels and alloys under static-load
conditions in high-temperature water**

*Corrosion des métaux et des alliages — Méthode d'essai pour le
mesurage de la vitesse de propagation des fissures de corrosion sous
contrainte des aciers et des alliages dans des conditions de charge
statique dans de l'eau à haute température*





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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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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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 — Test method for measuring the stress corrosion crack growth rate of steels and alloys under static-load conditions in high-temperature water

1 Scope

This document specifies a test method for determining the stress corrosion crack (SCC) growth rate of steels and alloys under static-load conditions in high-temperature water, such as the simulated water environment of light water reactors. The crack length of the specimen is monitored by a potential drop method (PDM) during the test in an autoclave.

The test method is applicable to stainless steels, nickel base alloys, low alloy steels, carbon steels and other alloys.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 7539-6, *Corrosion of metals and alloys — Stress corrosion testing — Part 6: Preparation and use of precracked specimens for tests under constant load or constant displacement*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 7539-6 and the following apply.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

potential drop method

PDM

non-destructive method for measuring a crack length based on the change in the electric potential as a crack propagates in the presence of an applied DC or AC current

3.2

stress corrosion crack transitioning

SCC transitioning

use of cyclic loading at low frequency and with increasing hold time at maximum load in the test environment to promote a transition in the fracture surface morphology from a transgranular (TG) fatigue pre-crack to SCC, typically intergranular (IG) or interdendritic (ID) morphology for austenitic alloys

3.3

crack-tip re-activation loading

use of loading cycles to re-activate the tip of crack when crack retardation is observed under a static loading

3.4 initial crack length

a_0
<compact tension (CT) specimen> distance from the load line to the initial crack tip

Note 1 to entry: It can refer to the machined notch tip or the air fatigue pre-crack front in the specimen.

Note 2 to entry: For other fracture mechanics geometries, refer to ISO 7539-6. The crack length (a) is often expressed as a proportion of the distance from the load-line to the end of the specimen (W): a/W .

3.5 final crack length

a_f
distance from the load line to the final crack front at the end of the stress corrosion crack growth test, where the crack length is measured on the fracture surface of the specimen

3.6 flow stress at test temperature

σ_{flowT}
algebraic average of the yield stress (σ_{yT}) and the ultimate tensile strength (σ_{uT}) at the test temperature:

$$\sigma_{flowT} = (\sigma_{yT} + \sigma_{uT})/2$$

3.7 crack engagement

specimen thickness B where the stress corrosion crack has advanced

Note 1 to entry: It is expressed as a percentage.

3.8 average crack extension

A_1
average crack extension across the specimen thickness using a crack growth area or many equally spaced measurements of crack length (equally divided method)

3.9 average crack extension in crack engagement area

A_2
average crack extension based on the fraction of the specimen thickness where the stress corrosion crack has occurred

3.10 minimum crack extension

A_{min}
minimum extension of the stress corrosion crack in the specimen

3.11 maximum crack extension

A_{max}
maximum extension of the stress corrosion crack in the specimen

4 Principle of test

Stress corrosion cracking is a phenomenon in which a crack grows in an environment when stress is applied to a susceptible material. Thus, stress corrosion cracking is affected by three general factors: the material, stress and environment. The SCC growth rate is affected by the stress intensity factor, K_I . The SCC growth rate, da/dt , is defined as the time derivative of the crack length. While there is often no clear distinction between static loading and some very slowly increasing monotonically or cyclic loading, the primary interest in most SCC growth testing is the behaviour under static loading. By

applying a static load to a specimen with a crack at a known K_I and continuously measuring the crack length (a) using the PDM, the crack growth rate (da/dt) can be continuously obtained. Often the best insight into the effects of environment and temperature are obtained by making periodic changes while continuously measuring the SCC growth response.

This document specifies the preparation of specimens, the control of the testing environment, the method of transitioning from fatigue crack to SCC, and the determination of the growth rate of a crack using fracture mechanics specimens in high-temperature water environments, with an emphasis on light water reactors.

Although the minimum requirements and basic procedures of SCC growth rate testing in high-temperature water are summarized in this document, it should be noted that there are complex interdependencies of many influential parameters on stress corrosion cracking phenomena, and subtle variations in test conditions can have a major impact on the reproducibility and credibility of the data. Extensive efforts to obtain high-quality SCC growth rate data have been undertaken over the last four decades and many key issues must be understood and implemented (see References [1] to [3]). For example, reliable SCC transitioning prior to static loading is an essential element, and specific procedures have been developed to help achieve well-behaved response.

5 Specimen

5.1 Specimen orientation

The specimen orientation in the test material is designed in accordance with ISO 7539-6.

The relative orientation of the crack plane and growth direction in the test material shall be specified in relation to the product form (such as plate rolling direction or pipe longitudinal direction) and, if applicable, also specified in relation to the weld direction and additional cold work (e.g. for rolling or forging). When the specimen is taken in or near a weld, the location of the crack plane of the specimen in relation to the weld fusion line shall be provided because a very pronounced effect on SCC behaviour is expected when the crack in the specimen propagates in the heat affected zone or weld metal of the test material, and the properties can vary from the weld root to the weld crown.

5.2 Specimen geometry

Many specimen geometries have been used for crack growth testing (see ISO 7539-6). The most common specimen is a compact tension (CT) specimen with a side-groove design, shown in [Figure 1](#). The specimen thickness, B , is usually between $12,5 \times 10^{-3}$ m and $25,4 \times 10^{-3}$ m. Smaller or larger specimens are sometimes used but shall be justified from K -size criteria (see [5.4](#)). The specimen width, W , is typically two times the specimen thickness (B).

Side grooves on both sides of the specimen are recommended to help maintain in-plane crack growth, but are not obligatory. The depth of each side groove is typically 5 % of B , and they are typically hemispherical.

The more complex contoured double cantilever beam (CDCB) specimen is also used because the stress intensity factor is practically considered constant over a certain range of crack lengths under constant-load conditions. However, note that the criteria for a CT specimen given in [5.4](#) is not applicable to a CDCB specimen. Details for a CDCB specimen are given in [Annex A](#).

β is the factor related to the extent of plasticity considered acceptable ($\beta = 2,5$ is sometimes used);

K_I is the stress intensity factor (in MPa $\sqrt{\text{m}}$);

σ_{yT} is the yield stress at the test temperature (in MPa).

However, in [Formula \(1\)](#), when $\sigma_{uT} / \sigma_{yT} > 1,3$ is satisfied, σ_{flowT} can be used instead of σ_{yT} .

5.5 Specimen dimensional measurement

The dimensions of the specimen are measured and confirmed to be within the fabrication tolerance shown in [Figure 1](#).

5.6 Stress intensity factor, K_I

The stress intensity factor, K_I , for a CT specimen is defined as shown in [Formula \(2\)](#):

$$K_I = \frac{P}{B_e \sqrt{W}} \frac{(2+\alpha)}{(1-\alpha)^{3/2}} (0,886 + 4,64\alpha - 13,32\alpha^2 + 14,72\alpha^3 - 5,6\alpha^4) \quad (2)$$

where

K_I is the stress intensity factor (in MPa $\sqrt{\text{m}}$);

P is the load (in MN);

α a/W .

NOTE The accuracy of [Formula \(2\)](#) is $\pm 0,5$ % over the range $0,2 \leq \alpha \leq 1,0$. K_I can also be obtained by finite element analysis.

6 Test equipment

A typical test system consists of a high-temperature, high-pressure autoclave with a loading machine and a water circulation system that flows a simulated light water reactor environment through the autoclave. While the volume and pressure of the autoclave depend on the size of the specimen and target test temperature, a smaller volume is generally preferable to achieve better control of the test parameters. An ion exchanger shall be incorporated in the circulation loop to remove ionic impurities generated by corrosion of the test specimen or system materials. If dosing species (such as pH control additives or boric acid) are added to the test environment for a simulated pressurized water reactor (PWR) environment, the demineralizer shall be equilibrated to the desired chemistry (e.g. B, Li). A once-through system can also be used to maintain the environment chemistry, although a circulating system generally provides a better control of the water chemistry in the autoclave because of the higher refresh rate. For relatively pure boiling water reactor (BWR) environments, a recirculating system is necessary, and if impurities are desired (e.g. 30 $\mu\text{g/l}$ sulfate), they are ideally continuously added to the water flowing into the autoclave, with appropriate continuous monitoring of ionic species or solution conductivity, and clean-up of return water from the autoclave.

Control of dissolved gases, especially oxygen or hydrogen, in the test environment is usually accomplished by continuous bubbling in the reservoir, which ideally has a much smaller diameter than height, and generally should be of limited volume (e.g. < 10 l). The solubility of oxygen at standard temperature and pressure (STP) is about 43 mg/l (and varies with temperature), and the solubility of hydrogen is about 1,6 mg/l (and varies less near room temperature). Conditions (dissolved gases, autoclave volume, refresh rate and system materials) shall be chosen to ensure that the outlet gas concentration is a high fraction of the inlet (e.g. > 80 %); this is especially an issue for dissolved oxygen concentrations below about 100 $\mu\text{g/l}$, and a bigger issue if hydrogen is also present.

Test stability is important, and load, temperature, water purity and dissolved gas concentration shall be kept constant during a given test segment. Stability of room temperature is also important, because it can affect dissolved gas concentration, water purity and precision instrumentation used to control temperature and measure crack length.

The test equipment includes devices to continuously monitor the crack length, temperature, water quality parameters such as solution conductivity and dissolved gases, and corrosion potential. The corrosion potential measurement is less important in a simulated PWR environment (with dissolved hydrogen and no dissolved oxygen) than in a simulated BWR environment with dissolved oxygen. Nevertheless, measuring corrosion potential is recommended to ensure that the corrosion potential has reached an appropriate value prior to the testing and to detect changes in water chemistry.

Some examples of test equipment in BWR and PWR water environments are provided in [Annex B](#), together with a typical schematic diagram of the water loop. More detailed water chemistry and monitoring items are provided in [Annex C](#) and Reference [3].

7 Crack length measurement by potential drop method

7.1.1 Both direct current (DC) and alternating current (AC) PDMs have been used. DC is most widely used because it is less susceptible to electrical noise, and easier and less expensive to implement.

7.1.2 The PDM has many applications in the measurement of the fatigue crack growth rate and extensive guidelines exist for measuring fatigue crack growth rates (see References [8] and [9]). While the majority of the practices described in the standards are valid when applied to SCC growth testing, there are additional factors specific to SCC growth tests in high-temperature water environments.

7.1.3 For high-quality PDM measurements using reversed DC current, a digital voltmeter capable of integration over ≥ 1 power line cycle is essential, and many \pm current readings shall be averaged (typically 100 to 5 000, depending on the crack growth rate) to achieve good crack length resolution. If PDM readings are taken during any unloading and reloading cycles, some bias downward in crack length may be observed from crack closure.

7.1.4 The wires inside the high-temperature autoclave shall be insulated. Below about 300 °C, polytetrafluoroethylene (PTFE) (standard tubing, or heat-shrinkable tubing) is commonly used. At higher temperatures, pieces of partially stabilized zirconia (e.g. 3 % mass fraction MgO) are commonly used.

7.1.5 The electrical resistivity changes with time at temperature in many nickel base alloys, and can create the false impression of crack advance when using PDM measurements. Higher temperature or cold work produces a faster approach to saturation. The need for compensation for resistivity changes depends on both the alloy and heat as well as the crack growth rate being measured. For example, at 10^{-8} m/s, a resistivity change even early in the test would not affect the subsequent measured growth rate. But below 10^{-11} m/s, small changes can affect the measured growth rate. A "reference potential" shall be measured on the same material and condition as the specimen because the alloy, heat, cold work and prior temperature exposure affect the resistivity change versus time. The reference potential can be measured on a separate rod or coupon, or on the back-face of the specimen. When the back-face potential is used, the change in reference potential as the crack grows should be considered. The resistivity change with time for stainless and ferritic steels is much smaller than nickel base alloys, and compensation is rarely done. Historically, a reference potential measurement has been used to compensate for temperature or DC current fluctuations, but eliminating these fluctuations is much more effective than compensating for them.

7.1.6 The temperature fluctuation in the autoclave has a big effect on the accuracy of the crack length measurement by the PDM. It is necessary to suppress the change of temperature as much as possible.

7.1.7 Uneven crack advance is more common in SCC growth tests in high-temperature water than in fatigue tests, and it significantly affects the accuracy of all forms of crack length measurement, which

are strongly biased by the regions of least crack advance. The (commonly IG) crack morphology, crack branching and less planar crack growth in SCC versus fatigue growth tests can produce current shorting in the wake of the crack, causing a significant underestimation of crack size. In such circumstances, where a large discrepancy is observed between the PDM and fractography measurements, a linear post-test correction of crack depth, crack growth rate and K is recommended because it is not known how or when the problems developed during the test.

The following issues should be addressed to reduce the noise of the PDM signal: wire routing of the PDM cables, electrical insulation between the specimen(s) and the loading clevis, position and stability of the current lead attachment.

The current applied to the specimen during PDM measurements can affect the measurement of corrosion potential, although the effect is minimized by using continuous PTFE insulation of the current leads from outside the autoclave to the specimen. It is recommended that the current applied to the specimen be turned off for several seconds before corrosion potential measurements.

8 Corrosion potential measurement

8.1 General

Corrosion potential (E_{corr}) shall be measured in simulated BWR environment tests. It should be measured in simulated PWR environment tests.

8.2 Measurement method

A high-input-impedance ($\geq 10^{14} \Omega$ is recommended) electrometer is used to measure the potential between the CT specimen and a reference electrode.

Several types of reference electrodes are commonly used:

- Ag/AgCl/Cl⁻ electrodes (internal type and pressure balanced external type);
- Membrane, metal/metal oxide electrodes (Fe/Fe₃O₄, Cu/Cu₂O, etc.).

Also, to ensure the accuracy of the potential measurements, including a platinum electrode is recommended because its potential is precisely known in hydrogen-only environments, and is quite well defined in oxygen-only environments.

9 Test procedure

9.1 General

A flow chart of SCC growth testing is shown in [Figure 2](#). After preparing the specimen and getting ready for the PDM measurement and E_{corr} measurement (leads, reference electrode, etc.), the crack growth test is performed using the following procedures.

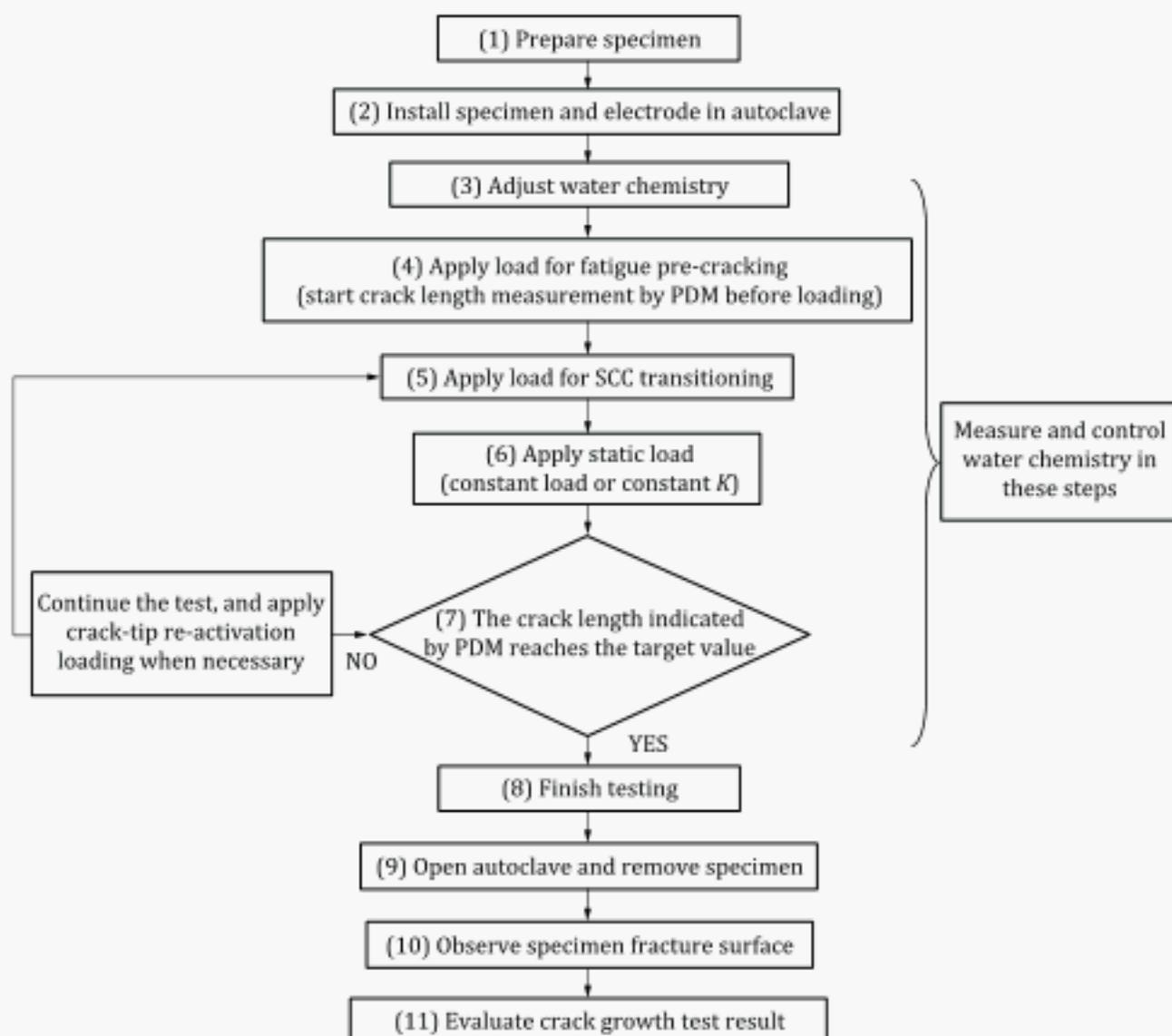


Figure 2 — A flow chart of SCC growth test in simulated light water reactor environment

9.2 Installation in autoclave

9.2.1 After cleaning, the specimen is installed in the loading clevises, and leads for PDM are attached: one set for the DC or AC current to the specimen and one for measuring the electrical potential drop as the crack grows. A third set is needed if reference potentials are used. The loading linkage shall be insulated at least at one location to eliminate an alternate current path. The current shall flow only through the specimen (see 7.1.4). A ground isolated power supply is also essential to prevent "ground loops" (current that could flow through the linkage and autoclave materials to ground, thereby creating an alternative path to its flow through the specimen).

9.2.2 A reference electrode for the E_{corr} measurement is installed in the autoclave near the specimen. Also, it is recommended that a platinum electrode is installed near the reference electrode. The wiring and inter-connections (which are often a limiting factor) shall be carefully designed to maintain a high impedance between the two leads.

9.3 Adjustment of test environment

9.3.1 The water chemistry in the autoclave is limited by the water quality in the reservoir, the autoclave volume and the refresh rate. At a minimum, it shall be characterized by monitoring the autoclave inlet and outlet solution conductivity and dissolved oxygen.

9.3.2 After preparing pure water using ionic exchange resins, the solution conductivity at room temperature (25 °C) should be < 6 µS/m (which is affected by exposure to air). Then, the water chemistry condition is adjusted to the test conditions, e.g. simulated BWR or PWR water. For simulated PWR primary environments, the concentrations of boric acid and lithium (or potassium) hydroxide are controlled. Examples of the water chemistry conditions for simulated BWR and PWR environment tests are shown in [Annex C](#).

Note that mixed bed demineralizers often initially contain high levels of organics, and most have no effect on solution conductivity until they are heated to 300 °C and decompose, and thus affect the outlet solution conductivity and the environment to which the specimen is exposed. Pre-cleaning of new demineralizers for 4 to 8 days using a benchtop loop comprised of a closed beaker (to minimize air exposure), a small pump, the demineralizer, a sub-micron filter and a UV light is recommended.

9.3.3 The dissolved oxygen and hydrogen concentrations are adjusted by bubbling gases (O₂, H₂, N₂, Ar, etc.) in the reservoir. During the test, the water chemistry parameters, such as gas concentration, solution conductivity, ion concentration and temperature, shall be controlled within the desired ranges. The refresh rate of water through the autoclave shall be sufficient to ensure that the water chemistry at inlet and at outlet of the autoclave is high quality and stable during the test. The parameters typically used in simulated BWR and PWR tests are shown in [Annex C](#).

9.3.4 When multiple specimens are tested in an autoclave, the temperature difference between them should be characterized and minimized (ideally < 1 °C).

9.3.5 Temperature fluctuations in the autoclave have a big effect on the accuracy and resolution of crack length measurements by PDM, and they should be minimized (ideally < 0,2 °C).

9.4 Loading

9.4.1 General

The specimen is loaded after the PDM system is operating and the water chemistry is stable (as described in [9.3](#)) and sufficient time has passed for development of the oxide film (generally a few days). In water with oxygen, the corrosion potential will usually continue to rise slowly for a week or two.

9.4.2 Fatigue pre-cracking

A fatigue pre-crack shall be introduced before SCC transitioning described in [9.4.3](#).

The final maximum load during pre-cracking shall not exceed the initial load used for the SCC growth test because compressive plastic strain at the crack tip due to a decrease in applied load can have a significant effect on the applied K_I for the SCC growth test.

The fatigue pre-crack can be introduced in air or in the test environment. Familiarity with ISO 11782-2^[10] and ASTM E647-15e1^[8] is recommended.

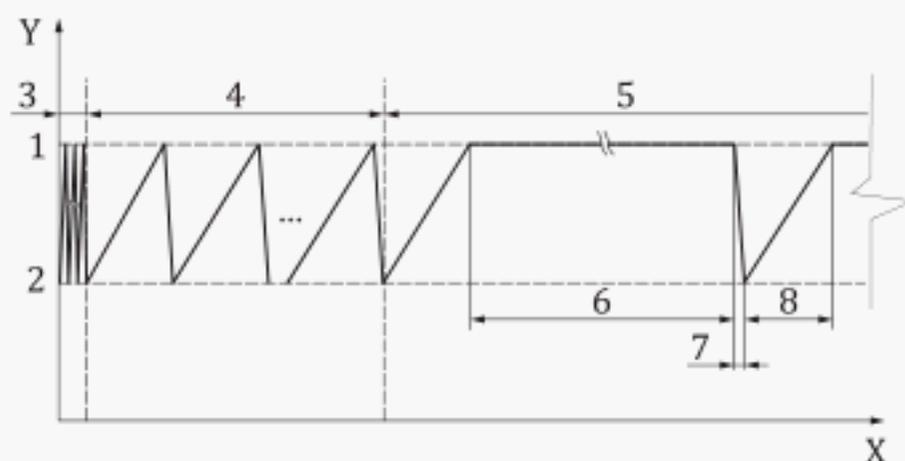
9.4.3 SCC transitioning

Starting SCC growth tests under a static loading condition immediately after the TG fatigue pre-crack often results in no or little growth of IG or ID SCC from isolated multiple areas along the fatigue crack front especially in relatively resistant material to stress corrosion cracking. IG/ID SCC usually propagate unevenly, especially when areas of the fatigue crack front are pinned and no IG growth occurs in those

areas. This causes large uncertainties in the applied K_I and the DC potential drop indicated crack length (and therefore growth rate).

Since a TG fatigue pre-crack essentially never exists in plant components, a complete transition to IG or ID SCC (100 % SCC engagement) for austenitic alloys is very important for obtaining relevant and reproducible SCC growth rates.

The transitioning to SCC is accomplished by obtaining sustaining crack advance while reducing cyclic contribution by decreasing the frequency, then increasing the hold time at maximum load. A common approach is to cycle at about 0,01 Hz at $R = 0,5$, then reduce the frequency to 0,001 Hz keeping the maximum load (P_{max}) or K_I ($K_{I,max}$) at or near the load used for SCC growth testing. Trapezoidal loading with increasing hold times at P_{max} or $K_{I,max}$ is then used to transition to static loading. Hold times at P_{max} or $K_{I,max}$ commonly used are ~3 h, ~9 h and ~24 h, but high growth rate materials and environments (e.g. $> 10^{-9}$ m/s) may require only a ~9 h hold time, and low growth rate situations (e.g. $< 10^{-11}$ m/s) may require four or five hold time steps. The example of applying load sequence for SCC transitioning is shown in Figure 3. It is important that the growth rate behaviour be well-behaved in each step, not decaying versus time.



Key

X	test time	4	step 2 (lower frequency, e.g. 0,001 Hz)
Y	load (P) or stress intensity factor (K)	5	step 3 (multi steps cycle including hold time)
1	P_{max} or $K_{I,max}$	6	hold time, T_h (e.g. 3 h, 9 h or 24 h)
2	P_{min} or $K_{I,min}$	7	fall time, T_f
3	step 1 (higher frequency, e.g. 0,01 Hz)	8	rise time, T_r

Figure 3 — Example of applying load sequence for SCC transitioning

It is recommended that a load ratio of $< 0,5$ be used for lower K_{max} tests (e.g. $< 20 \text{ MPa}\sqrt{\text{m}}$), and $> 0,7$ for high K_{max} tests (e.g. $> 60 \text{ MPa}\sqrt{\text{m}}$). When the growth rate is decaying versus time, a lower load ratio is often needed.

Since it is the rising part of cyclic waveform that enhances crack growth, an asymmetric waveform (e.g. 5 % fall, 95 % reload) is recommended during SCC transitioning.

References [3], [11], [12] and [13] provide more details on SCC transitioning in both unirradiated and irradiated materials.

9.4.4 Static loading

9.4.4.1 Constant load

A constant load condition is commonly used in a static-load SCC growth test. When applying a constant load to a CT specimen, the K_I of the specimen increases slowly with crack advance. As the crack becomes longer, the rate of K increase is faster. When the amount of crack advance during the test is large, it is necessary to take the increase in K and its increasing rate into consideration in the test result evaluation.

9.4.4.2 Constant K

The effect of the increasing K during testing can be reduced by constant- K tests. A typical constant- K test is performed by shedding the load applied to a CT specimen as the crack grows so that the applied K_I calculated based on the PDM signal stays essentially constant. Although the error in the crack size measurement by PDM results in some variation in K , the effect of the change in K is usually smaller than constant load tests.

Very small changes in applied load are preferred, but this is controlled by crack length resolution. The magnitude of each load correction shall be limited so that one unusual PDM reading does not produce over-correction. It is recommended that apparently shortening of the crack not result in an increase in load, to avoid hunting or load cycling.

An alternative way to achieve a constant K condition is to use a CDCB specimen. As noted in 5.2, the K_I of a CDCB specimen remains almost constant as the crack advances with a fixed applied load.

9.4.4.3 Changes in loading condition

Lowering the applied load by more than ~1 % during testing is not recommended since it can leave a compressive stress field at the crack tip. When increasing the applied load, small increments of a few per cent are recommended. For both falling and rising load, additional changes can be made after sufficient crack advance. It must be recognized that K changes in most structural components occur because the crack has grown; that is, changes occur by dK/da , not by dK/dt or simply a sudden change in K .

When there is no significant increase in crack length over the duration of the SCC growth test segment, crack-tip re-activation using a slow cyclic load is considered appropriate, with subsequent transitioning as described in 9.4.3. When a test must be interrupted, the test should be restarted in accordance with the procedures described in 9.4.1.

9.4.5 It should be noted that the crack front unevenness significantly affects the accuracy of PDM measurement, with regions of least crack advance strongly biasing PDM. This in turn affects the indicated crack length, crack growth rate and K values. While unevenness is common in components or specimens with extensive SCC growth, the most reliable data are obtained when unevenness is small (e.g. < 10 or 20 % of the SCC growth), in part because PDM does not reflect an average crack advance (and there is no way to know how the unevenness evolved during a test), and in part because regions of least crack advance can begin to grow as the stress on those areas rises, which can give the impression of rapid growth even though it is occurring in a relatively small, high stress area. Both test design and test management are needed to minimize this concern: excellent SCC transitioning procedures are needed to achieve 100 % SCC engagement from the fatigue pre-crack, and consideration shall be given to the subsequent development of unevenness after extensive SCC growth. For example, after several millimetres of crack growth, cyclic loading can be used to straighten the crack front and permit evaluation of other areas of the microstructure.

10 Evaluation of test results

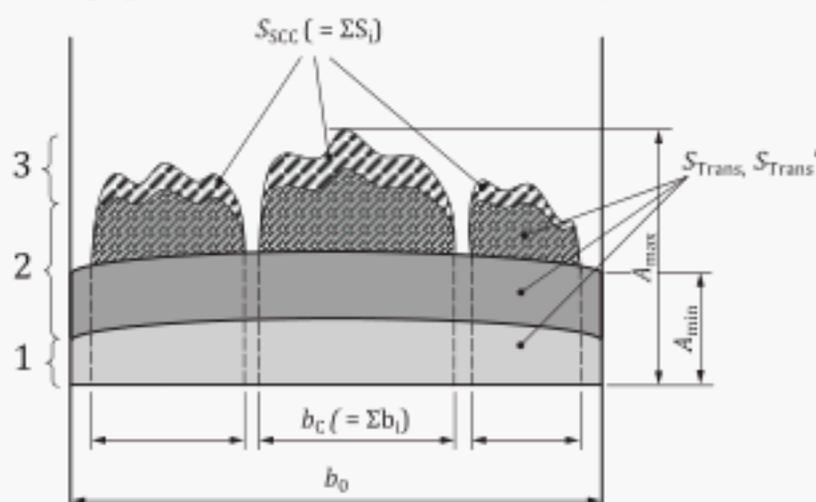
10.1 When the test is complete, the specimen is fractured apart by fatigue in air. The fatigue pre-crack and the SCC growth surface developed in the high-temperature high-pressure water environment can

be observed with an optical microscope (where the oxide formed in high-temperature water is usually visible) and/or scanning electron microscope (SEM), and the images recorded. The detailed fracture morphology in the SCC region should be observed by SEM and the image recorded.

10.2 The schematic fracture surface of the specimen of austenitic alloy is shown in [Figure 4](#). The crack extensions A_1 (averaged over specimen thickness), A_2 (averaged over SCC engaged area) and A_{\min} / A_{\max} (minimum and maximum crack extension) are determined from the fracture surface of the specimen as shown in the figure, for example.

10.3 The average crack extension values can be determined by the area method or equally divided method, as shown in [Figures 4](#) and [5](#), respectively. When the unevenness of the crack is significant, a higher number of divisions is necessary (see [Figure 5](#)).

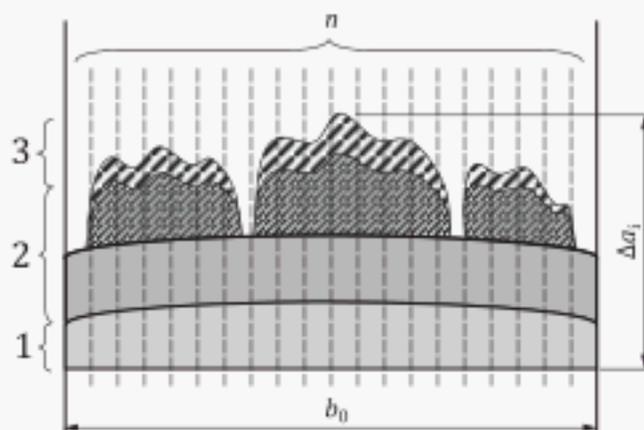
For a highly uneven, ID crack morphology, such as often develops in nickel base weld metals, the procedure to correct crack length and crack growth rate using crack engagement is described in the literature (see Reference [\[14\]](#)). However, the objective should always be to achieve 100 % engagement.



Key

1	fatigue pre-crack (TG)	b_0	thickness
2	SCC transitioning (TG to IG)	b_c	crack width
3	SCC growth region under static loading (IG)	A_{\max}	maximum crack extension
S_{SCC}	SCC growth area under static loading	A_{\min}	minimum crack extension
S_{Trans}	transitioning area for fatigue pre-cracking and SCC transitioning (excludes fatigue pre-crack in air)	A_1	average crack extension: $(S_{\text{SCC}} + S_{\text{Trans}}) / b_0$
$S_{\text{Trans}'}$	transitioning area corresponding to SCC engaged width (excludes fatigue pre-crack in air)	A_2	average crack extension in crack engagement area: $(S_{\text{SCC}} + S_{\text{Trans}'}) / b_c$

Figure 4 — Method for obtaining crack lengths on the fracture surface of specimen [area method]

**Key**

1	fatigue pre-crack (TG)	n	division number
2	SCC transitioning (TG to IG)	Δa_1	crack extension
3	SCC growth region under static loading (IG)	A_1	average crack extension: $\Sigma \Delta a_1 / n$
b_0	thickness		

Figure 5 — Method for obtaining crack lengths on the fracture surface of a specimen [equally divided method]

10.4 The crack lengths obtained throughout the test by PDM measurement are corrected based on the initial crack length (a_0) and the final crack length (a_f) that are determined from A_1 and A_2 (or, in rare cases, A_{max}), as described in 10.2. A_2 is most commonly used for correction because it is considered unlikely that regions of higher susceptibility (reflected in the regions of deeper cracking) extend throughout a structural component.

When the corrected crack length versus time response is linear with time in the evaluation period and the slope is significant compared to the PDM data fluctuations (e.g. standard deviation, σ), the crack growth rate, da/dt , is calculated.

The schematic figure of the method to determine the crack growth rate from the crack length versus test time is provided in Annex D.

NOTE When the water chemistry or the test load is changed during the SCC growth test, the SCC growth rate is evaluated from the PDM signal during which the test conditions are stable.

10.5 The stress intensity factor, K_I , during the SCC growth rate evaluation period is determined from the corrected crack length and the applied load by the method described in 5.6. When the change in the stress intensity factor during the segment is significant, the averaged value should be used, and the range reported.

10.6 The quality of the SCC growth rate shall be evaluated in terms of whether it is well-behaved, linear and reproducible. It is also important to evaluate the actual experimental conditions (water chemistry, loading, etc.) during the test based on the following considerations:

- stability of test conditions;
- evenness of crack front;
- crack engagement;
- linearity of the change in crack length versus time (linear, rising, decaying, etc.) and its response to changes in water chemistry, temperature, loading, etc.;

- e) post-test crack length correction, which is represented by a ratio of actual crack growth increment to crack growth increment measured by PDM;
- f) specimen K -size requirements given in 5.4.

The minimum requirements for qualifying crack growth rate data sometimes depend on the application of the data, and discussions among the interested parties is encouraged. References [3] and [15] are helpful for evaluating crack growth rate data.

11 Test report

The test report should include the following information:

- a) type of material;
- b) chemical composition;
- c) heat treatment conditions;
- d) mechanical properties of material (yield stress at test temperature, tensile strength at test temperature) (in MPa);
- e) geometry and dimensions of specimen (in m);
- f) orientation of the specimen including product form of the material, any added cold work, or heat-affected zone of welding;
- g) conditions used for fatigue pre-crack introduction [the maximum load (in MN), stress ratio, frequency (in Hz), load waveform, pre-crack length (in m), etc.];
- h) SCC test conditions, including:
 - 1) load (in MN) or stress intensity factor (in $\text{MPa}\sqrt{\text{m}}$);
 - 2) water chemistry conditions in simulated BWR environments: temperature (in °C), solution conductivity (inlet/outlet) (in mS/m), dissolved oxygen concentration (inlet and outlet) (in $\mu\text{g/l}$), corrosion potential (V versus SHE), sulfate ion concentration (in $\mu\text{g/l}$), chloride ion concentration (in $\mu\text{g/l}$);
 - 3) water chemistry conditions in simulated PWR environments: temperature (in °C), solution conductivity (inlet) (in mS/m), pH, boric acid concentration (in mg/l), lithium concentration (in mg/l), dissolved oxygen concentration (in $\mu\text{g/l}$), dissolved hydrogen concentration (in cm^3/kg), sulfate ion concentration (in $\mu\text{g/l}$), fluoride ion concentration (in $\mu\text{g/l}$), chloride ion concentration (in $\mu\text{g/l}$);
- i) SCC growth surface information [crack shape, crack engagement (in %), crack lengths shown in 10.2 (A_1 , A_2 , A_{min} and A_{max}), crack type];
- j) tabulated data versus time during the test (crack length, load, solution conductivity, dissolved oxygen concentration, etc.);
- k) plots of corrected crack length versus time;
- l) crack extension during each segment corresponding to loading mode applied during the test (transitioning, constant load/constant K , etc.) and environment;
- m) relation between crack growth rate (in m/s) and controlled variations in test conditions (e.g. stress intensity factor, water chemistry, temperature);
- n) SCC transitioning conditions, crack-tip re-activation conditions;
- o) final crack length a_f (in m), $W-a$ (in m), judgment of validity as described in 10.6;

- p) SCC growth rate (in da/dt) (in m/s) and the period used for crack growth rate evaluation;
- q) special issues during testing (e.g. unplanned test interruption or conditions out of specification).

Annex A (informative)

CDCB specimen geometry and stress intensity factor calculation

The contoured double cantilever beam (CDCB) specimen ([Figure A.1](#)), originally proposed by Mostovoy et al.^[16], is a design in which K_I is determined by the load independently of a/W . This specimen has a constant energy release rate, G , regardless of the value of a/W , a result of its varying height versus a/W . In general, the energy release rate of a cantilever beam is given by [Formula \(A.1\)](#). In a CDCB specimen, the rate of change of compliance, C , with respect to a/W is constant.

$$\begin{aligned} G &= \frac{K^2}{E} (1-\nu^2) \\ G &= \frac{P^2}{2B} \frac{\partial C}{\partial a} \\ C &= \frac{8}{EB} \int_0^a \left(\frac{3x^2}{h^3} + \frac{1}{h} \right) dx \\ m &= \left(\frac{3a^2}{h^3} + \frac{1}{h} \right) \end{aligned} \tag{A.1}$$

where

G is the energy release rate;

E is Young's modulus;

ν is Poisson's ratio;

P is the load;

C is the compliance;

B is the specimen thickness;

h is the height of the cantilever beam;

a is the crack length;

m is the shape factor.

The size of the specimen can be determined using a suitable m value consistent with the objectives of the test, the size of the material, the maximum loading capacity of the system, etc. [Formula \(A.1\)](#) is a theoretical formulation for an infinite-length specimen and, therefore, the formula only approximates an actual specimen of a finite length. It is necessary to correct m in accordance with the actual K_I and to confirm the range over which the variation of dK_I/da is sufficiently small by measuring C or obtaining it by finite element analysis.

Variation in the specimen geometry shown in [Figure A.1](#) can be used when the a/W range is identified over which K_I is constant. This is typically confirmed by measurements of C or by finite element analysis. Reference [17] investigated the applicability of some of the specimen types shown in [Figure A.1](#) by a fatigue crack growth test.

The stress intensity factor, K_I , for the CDCB specimens shown in [Figure A.1](#) are as follows.

a) K_I for a Type I CDCB specimen is given as shown by [Formula \(A.2\)](#):

$$K_I = \frac{P}{20} (0,075\ 8a^2 - 3,684\ 2a + 90,833) \quad (\text{A.2})$$

where

K_I is the stress intensity factor (in MPa $\sqrt{\text{m}}$);

P is the load (in kN);

a is the crack length (in mm).

NOTE 1 The valid range of [Formula \(A.2\)](#) is $23 \leq a \leq 33$ (in mm).

b) K_I for a Type II CDCB specimen is given as shown by [Formula \(A.3\)](#):

$$K_I = \frac{P}{15,9} (0,021\ 5a^2 - 0,926\ 6a + 44,854) \quad (\text{A.3})$$

where

K_I is the stress intensity factor (in MPa $\sqrt{\text{m}}$);

P is the load (in kN);

a is the crack length (in mm).

NOTE 2 The valid range of [Formula \(A.3\)](#) is $21,3 \leq a \leq 33,0$ (in mm).

c) K_I for a Type III CDCB specimen is given as shown by [Formula \(A.4\)](#):

$$K_I = 1,447\ 04P \quad (\text{A.4})$$

where

K_I is the stress intensity factor (in MPa $\sqrt{\text{m}}$);

P is the load (in kN);

a is the crack length (in mm).

NOTE 3 The valid range of [Formula \(A.4\)](#) is $22 \leq a \leq 55$ (in mm).

d) K_I for a Type IV CDCB specimen is given as shown by [Formula \(A.5\)](#):

$$K_I = 2,754\ 4P \quad (\text{A.5})$$

where

K_I is the stress intensity factor (in MPa $\sqrt{\text{m}}$);

P is the load (in kN);

a is the crack length (in mm).

NOTE 4 The valid range of [Formula \(A.5\)](#) is $22 \leq a \leq 25$ (in mm).

e) K_I for a Type V CDCB specimen is given as shown by [Formula \(A.6\)](#):

$$K_I = \frac{1,16P}{4,3} (25,272 - 1,174a + 0,0635a^2) \quad (\text{A.6})$$

where

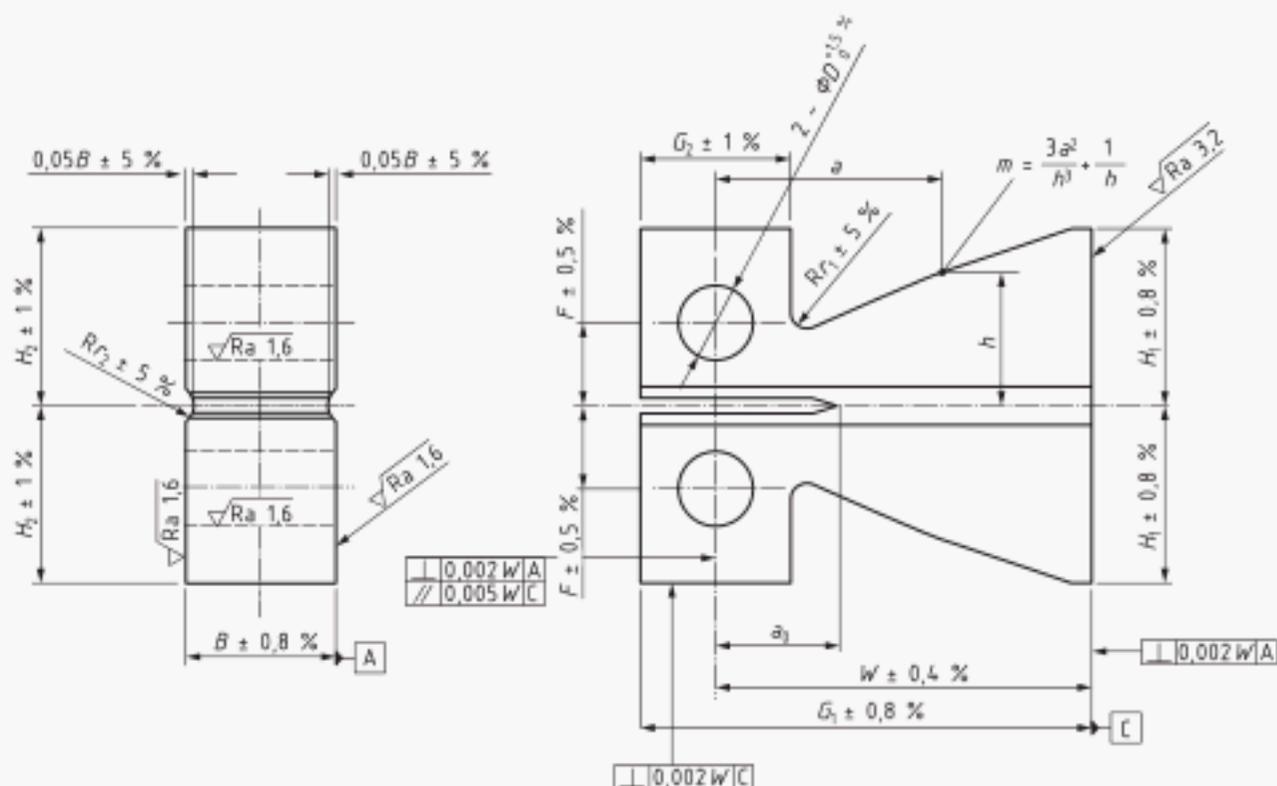
K_I is the stress intensity factor (in MPa $\sqrt{\text{m}}$);

P is the load in (kN);

a is the crack length (in mm).

NOTE 5 The valid range of [Formula \(A.6\)](#) is $9,5 \leq a \leq 13,5$ (in mm).

Because of its varying height, the CDCB specimen has the smallest sectional area between the loading axis and the crack tip. When the applied load is large, it is necessary to confirm that the plasticity does not significantly affect K_I . For example, an attempt has been made to determine the maximum applied load based on the confirmation of the linear relationship between the load increment and K_I by elastic-plastic finite element analysis.

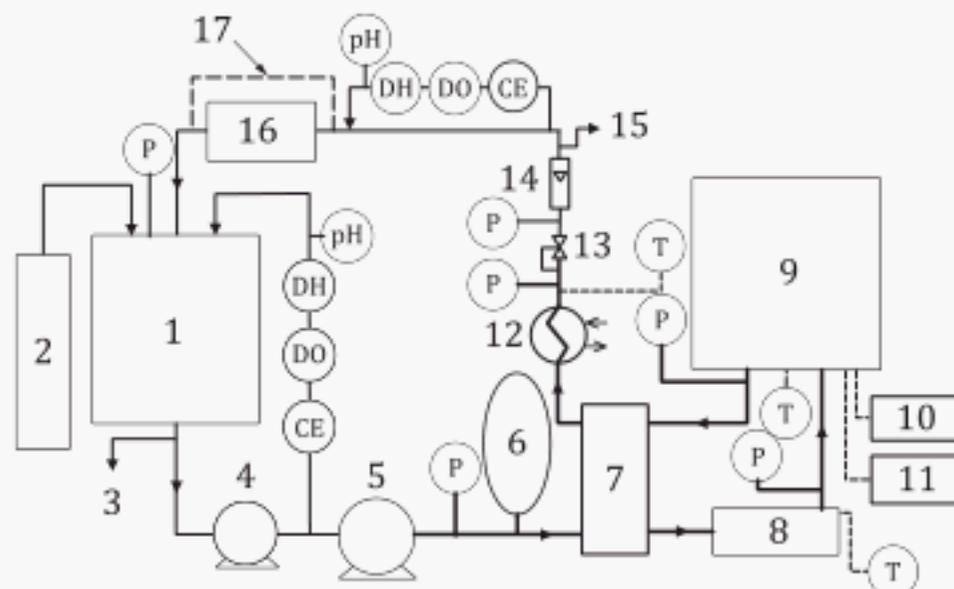


Type	B (mm)	W (mm)	G_1 (mm)	G_2 (mm)	H_1 (mm)	H_2 (mm)	F (mm)	D (mm)	r_1 (mm)	r_2 (mm)	m
I	25,4	60,0	70,0	22,0	28,27	30,0	17,0	12,7	2,5	4,0	0,437
II	25,4	63,5	76,2	25,4	30,0	30,0	17,0	12,7	3,0	4,0	0,44
III	25,4	91,6	104,3	27,7	50,0	32,5	21,0	12,7	2,0	4,0	0,20
IV	16,0	60,0	70,0	20,0	33,12	30,0	17,0	12,7	2,5	4,0	0,28
V	12,7	30,0	35,0	11,0	16,56	15,0	8,5	6,35	1,5	2,0	0,560

Figure A.1 — Schematic illustration and tabular dimensions for CDCB specimens

Annex B (informative)

Equipment for SCC growth testing



Key

1	water-chemistry-controlling reservoir	10	crack-length-measuring device
2	regulating gas injection system	11	corrosion-potential-measuring device
3	inlet water sampling point	12	cooler
4	feed water pump	13	pressure-regulating valve
5	high-pressure pump	14	flow meter
6	accumulator	15	outlet water sampling point
7	heat exchanger	16	ion exchange resin
8	preheater	17	bypass line
9	crack growth test equipment (autoclave with loading mechanism)		
P	pressure meter	DO	dissolved oxygen analyser
T	thermocouple	DH	dissolved hydrogen analyser
CE	solution conductivity meter	pH	pH meter

Bold lines indicate a high-pressure section.

Dashed lines represent electrical instrumentation.

Figure B.1 — Schematic diagram of test equipment (example)

Table B.1 — Basic specifications of simulated BWR environmental test equipment

Items	Basic specifications of test equipment
Main components of test equipment	Autoclave with a loading mechanism and low friction sliding seal, reservoir with controlled water chemistry, high-pressure pump, accumulator, heat exchanger, pre-heater, cooler, pressure-regulating valve, ion-exchange resin, regulating gas injection system, water chemistry-measuring devices, crack-length measuring device, E_{corr} -measuring device.
Load control	<p>A cyclic load is used for transitioning (fatigue pre-cracking to SCC transitioning) and for crack-tip re-activation, and the loading mechanism shall provide the following loading capability:</p> <ul style="list-style-type: none"> — load waveform: trapezoidal wave, sine wave (above 0,1 Hz); — frequency: 0,01 Hz to 0,000 1 Hz (~0,5 Hz for fatigue pre-cracking). <p>Because the internal pressure acts on the pull rod, the compensation of internal pressure shall be considered when the load is applied to the specimen. The friction between the sliding seal and the pull rod shall be confirmed to be low.</p>
Control of test pressure and test temperature	In a highly pressurized system, the over-pressure (relative to the vapour pressure of water at the test temperature) should be set so that no boiling occurs. Pressure sensors should be installed to monitor the pressure, and thermocouples in the autoclave near the specimen and near the top of the autoclave should be used to monitor temperature.
Measurement of test water chemistry and recording of test conditions	<p>Instruments should be continuously measured in the water at the inlet and outlet of the autoclave, including:</p> <ul style="list-style-type: none"> — Solution conductivity (at 25 °C): AC bipolar system; — Dissolved oxygen concentration (at 25 °C): Polarographic sensors or optical methods; <p>Other measurements as needed, potentially including:</p> <ul style="list-style-type: none"> — Dissolved hydrogen concentration (diaphragm electrode method or polarographic sensors); — pH: glass pH electrode method. <p>Sample points for the inlet and outlet water should be provided for water chemistry analysis.</p> <p>Quantitative analyses of inorganic ions contained as impurities in the inlet and outlet water should be performed (especially if the solution conductivity is elevated), including:</p> <ul style="list-style-type: none"> — Sulphate ion concentration: Ion chromatography method; — Chloride ion concentration: Ion chromatography method. <p>The following should be recorded to ensure the stability of the test conditions (both on-line and off-line recording systems can be used): Load, temperature, solution conductivity, dissolved oxygen concentration, dissolved hydrogen concentration (if necessary), E_{corr}.</p>

Table B.2 — Basic specifications of simulated PWR environmental test equipment

Items	Basic specifications of test equipment
Main components of test equipment	Autoclave with a loading mechanism and low friction sliding seal, reservoir with controlled water chemistry, high pressure pump, accumulator, heat exchanger, pre-heater, cooler, pressure-regulating valve, flow meter, inlet and outlet water sampling points, ion-exchange resin, gas bubbling system, water-chemistry-measuring device, crack-length-measuring device.
Load control	<p>A cyclic load is used (fatigue pre-cracking to SCC transitioning) and for crack-tip re-activation loading; therefore, a loading mechanism that can provide the following loading modes is needed:</p> <ul style="list-style-type: none"> — Load waveform: trapezoidal wave, sine wave (above 0,1 Hz); — Frequency: 0,01 Hz to 0,000 1 Hz (~0,5 Hz for fatigue pre-cracking). <p>Because the internal pressure acts on the pull rod, the compensation of internal pressure shall be considered when the load is applied to the specimen. The friction between sliding seal and the pull rod shall be confirmed to be low.</p>
Control of test pressure and test temperature	In a highly pressurized system, the over-pressure (relative to the vapour pressure of water at the test temperature) should be set so that no boiling occurs. Pressure sensors should be installed to monitor the pressure, and thermocouples in the autoclave near the specimen and near the top of the autoclave should be used to monitor temperature.
Measurement of test water chemistry and record of test conditions	<p>Instruments should be continuously measured in the water at the inlet and outlet of the autoclave, including:</p> <ul style="list-style-type: none"> — Solution conductivity (at 25 °C); — pH (at 25° C): Glass pH electrode; — Boric acid concentration: Neutralization titration; — Lithium ion concentration: Atomic absorption method; — Dissolved oxygen concentration (at 25 °C): Indigo carmine colorimetric method, dissolved oxygen meter (polarographic sensor) or optical sensor; — Dissolved hydrogen concentration: Dissolved hydrogen meter (diaphragm electrode method or polarographic sensor) or gas chromatography method; — Sulfate ion concentration: Ion chromatography method; — Fluoride ion concentration: Ion chromatography method; — Chloride ion concentration: Ion chromatography method; <p>Water inlet and outlet sample points shall be provided for water chemistry analysis.</p> <p>The Li and B concentrations define specific solution conductivity and pH values. Therefore, when impurity levels are low, the conductivity and pH measurements at RT can be used to determine the B and Li concentrations.</p> <p>The following data should be recorded to document the stability of the test conditions (both online and off-line recording systems can be used): load, temperature, solution conductivity, dissolved oxygen concentration, dissolved hydrogen concentration (if necessary), pH.</p>

Annex C (informative)

Water chemistry and monitoring items in simulated BWR and PWR environments

For BWR environments, both normal water chemistry (NWC) and hydrogen water chemistry (HWC) are often evaluated. An example of the preferred water chemistry for simulated BWR NWC conditions is shown in [Table C.1](#). It is important to maintain a low solution conductivity in the outlet water along with a low concentration of sulfate and chloride ions during the SCC growth rate test, unless the effect of impurities is being specifically investigated.

In PWR primary water, boron is added, as boric acid, to control the reactivity and LiOH is added to achieve the target pH at temperature (generally pH_T of 7,0 to 7,2 at 310 °C). Typical simulated PWR primary water conditions are shown in [Table C.2](#). Temperatures A, B and C for the PWR test provide the temperature at the outlet of the reactor pressure vessel, the outlet of the steam generator and in the pressurizer, 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.

It is recommended that the water chemistry items shown in [Table C.3](#) are measured during the test with adequate frequency. The timing of each measurement is classified into categories A, B and C. There are differences in some of the measured items for the simulated BWR environment test and the simulated PWR environment test.

Table C.1 — An example of water chemistry for testing in simulated BWR, NWC environment

Analysis items	Controlled values for water chemistry
Temperature	288 °C
Solution conductivity (outlet)	$\leq 8 \mu\text{S/m}$ ($\leq 0,08 \mu\text{S/cm}$) (at 25 °C)
Dissolved oxygen concentration	$> 200 \mu\text{g/l}$
Corrosion potential of austenitic CT specimen	$(0,15 \pm 0,05) \text{ V}$ versus SHE
Sulfate ion (SO_4^{2-}) concentration	$\leq 2 \mu\text{g/l}$
Chloride ion (Cl^-) concentration	$\leq 1 \mu\text{g/l}$

Table C.2 — Examples of water chemistry for testing in simulated PWR environment

Analysis items	Controlled values for water chemistry	
Temperature	Temperature A	325 °C
	Temperature B	290 °C
	Temperature C	343 °C
Key		
Temperature A: Temperature at the outlet of the reactor pressure vessel		
Temperature B: Temperature at the outlet of the steam generator		
Temperature C: Temperature in the pressurizer		
Environment I: Beginning of cycle chemistry		
Environment II: Middle of cycle chemistry		
^a Under STP (0 °C, 101,325 kPa).		

Table C.2 (continued)

Analysis items	Controlled values for water chemistry	
	Solution conductivity	Environment I
	Environment II	1,1 mS/m (11 µS/cm) (at 25 °C)
pH	Environment I	6,3 (at 25 °C)
	Environment II	6,65 (at 25 °C)
Boron concentration (as H ₃ BO ₃)	Environment I	1 500 mg/l
	Environment II	500 mg/l
Lithium concentration (as LiOH)	Environment I	3 mg/l
	Environment II	1 mg/l
Dissolved oxygen concentration	≤ 5 µg/l	
Dissolved hydrogen concentration	35 cm ³ /kg ^a	
Sulfate ion (SO ₄ ²⁻) concentration	≤ 20 µg/l	
Fluoride ion (F ⁻) concentration	≤ 20 µg/l	
Chloride ion (Cl ⁻) concentration	≤ 20 µg/l	
Key		
Temperature A: Temperature at the outlet of the reactor pressure vessel		
Temperature B: Temperature at the outlet of the steam generator		
Temperature C: Temperature in the pressurizer		
Environment I: Beginning of cycle chemistry		
Environment II: Middle of cycle chemistry		
^a Under STP (0 °C, 101,325 kPa).		

Table C.3 — Measurements during the test (example)

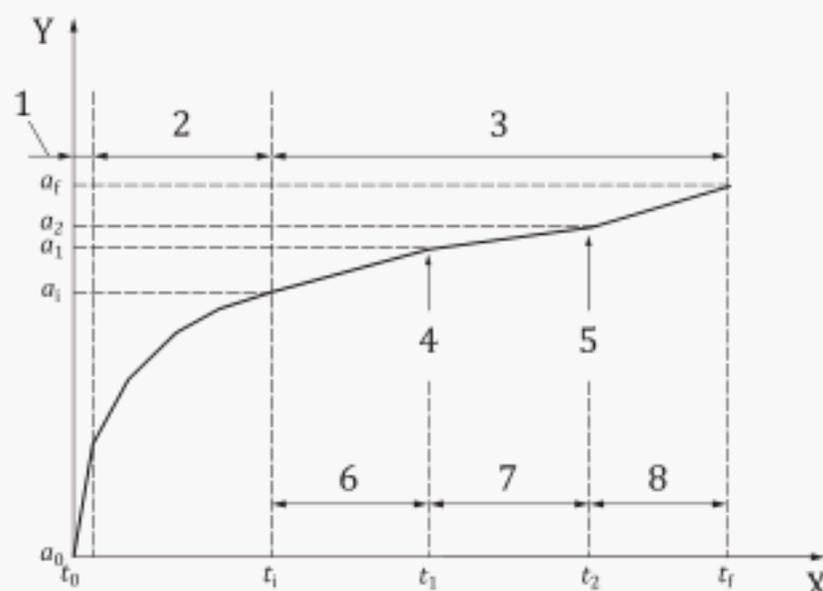
Measured items	BWR simulation condition	PWR simulation condition
Solution conductivity (inlet)	Category A	Category A
Solution conductivity (outlet)	Category A	Category B
Dissolved oxygen concentration (inlet)	Category A	Category B
Dissolved oxygen concentration (outlet)	Category A	Category B
Dissolved hydrogen concentration (inlet)	a	Category B
Dissolved hydrogen concentration (outlet)		Category B
Temperature	Category A	Category A
E_{corr}	Category A	a
Borate ion concentration	—	Category B
Lithium ion concentration	—	Category B
Sulfate ion (SO ₄ ²⁻) concentration	Category C	Category B
Chloride ion (Cl ⁻) concentration	Category C	Category B
Fluoride ion (F ⁻) concentration	a	Category B
pH	a	Category B
Key		
Category A: Continuously measured items		
Category B: Items measured at the beginning of the test, and at least once during the test		
Category C: Items measured at least once during the test		
^a Measured as necessary.		

Annex D (informative)

Approach to determine crack growth rate

The values of the crack length indicated by the PDM should be corrected to match the fractography based on incremental growth (initial crack length, a_0 , and the final crack length, a_f). Plots are then created to show the crack length versus test time, and the crack growth rate shown. The procedure to obtain the crack growth rate is given in Figure D.1. For example, the crack growth rate da/dt for segment I is the slope between time points t_1 and t_1 obtained by the least-squares method.

Because the crack growth rate may vary during a given segment (even if the test conditions are stable), the time period used for crack growth rate evaluation shall be carefully chosen and indicated.



Key

X	test time	4	water chemistry change
Y	crack length	5	water chemistry change
1	fatigue pre-cracking	6	segment I
2	SCC transitioning	7	segment II
3	static load (constant K or load)	8	segment III
a_0	initial crack length (distance from the load line to the initial crack front)	t_0	test time corresponding to a_0
a_f	final crack length (distance from the load line to the final crack front after the SCC growth test)	t_f	test time corresponding to a_f
a_1	crack length at the start point of segment I	t_1	test time corresponding to a_1
a_1	crack length at the start point of segment II	t_1	test time corresponding to a_1
a_2	crack length at the start point of segment III	t_2	test time corresponding to a_2

Figure D.1 — Schematic diagram of crack data, including SCC transitioning

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