INTERNATIONAL STANDARD

ISO 11782-2

First edition
1998-07-15

Corrosion of metals and alloys — Corrosion fatigue testing —

Part 2:
Crack propagation testing using precracked specimens

Corrosion des métaux et alliages — Essais de fatigue-corrosion —

Partie 2: Essais d'amorce de rupture sur des éprouvettes préfissurées

ISO 11782-2:1998(E)

Contents

Page

1 Scope **1**
2 Normative reference **1**
3 Definitions **1**
4 Test... **3**
  4.1 Principle of corrosion fatigue crack propagation testing ... **3**
  4.2 Specimens for corrosion fatigue crack propagation testing **4**
5 Apparatus **5**
6 Fatigue precracking **6**
7 Test conditions **7**
8 Test procedure **9**
9 Test report **11**
Annex A (informative) **Information on methods for measuring crack lengths** **13**

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

Printed in Switzerland

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11782-2 was prepared by Technical Committee ISO/TC 158, *Corrosion of metals and alloys*.

ISO 11782 consists of the following parts, under the general title *Corrosion of metals and alloys — Corrosion fatigue testing*:

— *Part 1: Cycles to failure testing*

— *Part 2: Crack-propagation testing using precracked specimens*

Annex A of this part of ISO 11782 is for information only.

Introduction

Crack propagation testing employs precracked specimens to provide information on the threshold conditions and on rates of corrosion fatigue crack growth. These data can be used in the design and evaluation of engineering structures where corrsion fatigue crack growth can dominate component life.

Because of the need to maintain elastically constrained conditions at the crack tip, the precracked specimens used for crack propagation tests are not suitable for the evaluation of thin products such as sheet or wire and are generally used for thicker products including plate, bar and forgings. They can also be used for parts joined by welding.

The results of corrosion fatigue testing are suitable for direct application only when the service conditions exactly parallel the test conditions especially with regard to material, environmental and stressing considerations.

INTERNATIONAL STANDARD © ISO **ISO 11782-2:1998(E)**

Corrosion of metals and alloys — Corrosion fatigue testing —

Part 2:
Crack propagation testing using precracked specimens

1 Scope

1.1 This part of ISO 11782 describes the fracture mechanics method of determining the crack growth rates of pre-existing cracks under cyclic loading in a controlled environment and the measurement of the threshold stress intensity factor range for crack growth below which the rate of crack advance falls below some defined limit agreed between parties.

1.2 This part of ISO 11782 provides guidance and instruction on corrosion fatigue testing of metals and alloys in aqueous or gaseous environments.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this part of ISO 11782. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this part of ISO 11782 are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 7539-1:1987, *Corrosion of metals and alloys — Stress corrosion testing — Part 1: General guidance on testing procedures.*

3 Definitions

For the purposes of this part of ISO 11782, the following definitions apply.

3.1 corrosion fatigue: Process involving conjoint corrosion and alternating straining of the metal, often leading to cracking.

NOTE — Corrosion fatigue may occur when a metal is subjected to cyclic straining in a corrosive environment.

3.2 force, P: Force applied to the specimen considered positive when its direction is such as to cause the crack faces to move apart.

3.3 maximum force, $P_{\max}$: Algebraic maximum value of force during a loading cycle.

3.4 minimum force, $P_{\min}$: Algebraic minimum value of force during a loading cycle.

3.5 force range, ΔP: Difference between the algebraic maximum and minimum values of the force.

3.6 stress intensity factor, K_I: Function of applied load, crack length and specimen geometry having dimensions of stress (length)$^{1/2}$ which uniquely defines the elastic stress field intensification at the tip of a crack subjected to opening mode displacements (mode I).

NOTE — It has been found that stress intensity factors, calculated assuming that specimens respond purely elastically, correlate the behaviour of real cracked bodies provided that the size of the zone of plasticity at the crack tip is small compared to the crack length and the length of the uncracked ligament. In this standard, mode I is assumed and the subscript I is implied everywhere.

3.7 maximum stress intensity factor, K_{max}, in fatigue: Highest algebraic value of the stress intensity factor in a cycle corresponding to the maximum load.

3.8 minimum stress intensity factor, K_{min}, in fatigue: Lowest algebraic value of the stressity factor in a cycle.

NOTE — This value corresponds to the minimum load when the stress ratio, R, is greater than zero and is set equal to zero when R is less than or equal to zero.

3.9 range of stress intensity factor, ΔK, in fatigue: Algebraic difference between the maximum and minimum stress intensity factors in a cycle:

$$\Delta K = K_{max} - K_{min}$$

3.10 threshold stress intensity factor range, ΔK_{th}, in fatigue: Value of the stress intensity factor range below which the rate of crack advance becomes insignificant for the application.

3.11 stress ratio, R, in fatigue loading: Algebraic ratio of the minimum and maximum force in a cycle

$$R = \frac{P_{min}}{P_{max}} = \frac{K_{min}}{K_{max}}$$

3.12 cycle: Smallest segment of the load- or stress-time function which is repeated periodically. The terms fatigue cycle, load cycle and stress cycle are also commonly used.

3.13 fatigue crack growth rate, da/dN: Rate of crack extension caused by fatigue loading and expressed in terms of crack extension per cycle.

3.14 stress intensity factor coefficient, Y: Factor derived from the stress analysis for a particular specimen geometry which relates the stress intensity factor for a given crack length to the load and specimen dimensions.

3.15 plane strain fracture toughness, K_{Ic}: The critical value of K at which the first significant environmentally independent extension of the crack occurs under the influence of rising stress intensity under conditions of high constraint to plastic deformation.

3.16 specimen orientation: The fracture plane of the specimen identified in terms of firstly the direction of stressing and secondly the direction of crack growth expressed with respect to three reference axes. These are identified by the letters X, Y and Z.

where

Z is coincident with the main working force employed during manufacture of the material (short-transverse axis);

X is coincident with the direction of grain flow (longitudinal axis);

Y is normal to the X and Z axes (see figure 1).

3.17 crack length, a**:** Effective crack length measured from the crack tip to either the mouth of the notch or the loading point axis depending on the specimen geometry.

3.18 specimen width, W**:** Effective width of the specimen measured from the back face to either the face containing the notch or the loading plane depending on the specimen geometry.

3.19 waveform: Shape of the peak-to-peak variation of load as a function of time.

3.20 cyclic frequency: Number of cycles per unit time, usually expressed in terms of cycles per second (Hz).

4 Test

4.1 Principle of corrosion fatigue crack propagation testing

A fatigue pre-crack is induced in a notched specimen by cyclic loading. As the crack grows the loading conditions are adjusted until the values of ΔK and R are appropriate for the subsequent determination of ΔK_{th} or crack growth rates and the crack is of sufficient length for the influence of the notch to be negligible.

Corrosion fatigue crack propagation tests are then conducted using cyclic loading under environmental and stressing conditions relevant to the particular application. During the test, crack length is monitored as a function of elapsed cycles. These data are subjected to numerical analysis so that the rate of crack growth, da/dN, can be expressed as a function of the stress intensity factor range, ΔK.

Crack growth rates presented in terms of ΔK are generally independent of the geometry of the specimen used. The principle of similitude allows the comparison of data obtained from a variety of specimen types and allows da/dN versus ΔK data to be used in the design and evaluation of engineering structures provided that appropriate mechanical, chemical and electrochemical test conditions are employed. An important deviation from the principle of similitude can occur in relation to short cracks because of crack-tip chemistry differences, microstructurally sensitive growth and crack tip shielding considerations.

The threshold stress intensity factor range for corrosion fatigue, ΔK_{th} may be higher or lower than the threshold in air depending on the particular metal/environment conditions. It may be determined by a controlled reduction in load range (see 6.3) until the rate of growth becomes insignificant for the specific application. Practically, from a measurement perspective it is necessary to assign a value to this (see 8.5).

NOTE — Both crack growth rate measurements and threshold stress intensity factor range determinations can be markedly affected by residual stresses. Thermal stress relief should, therefore, be considered prior to testing, but if this is not acceptable, the possibility of an effect should be recognized in the interpretation of the results. In particular, the presence of residual stresses can lead to an apparent dependence of ΔK_{th} on specimen thickness. Thickness effects can also arise in principle in relation to hydrogen charging and also where through-thickness transport of fluid occurs in flowing aqueous solutions. In the latter case it should be recognized that solution transport via the crack sides in the through-thickness direction is an artifact of the fracture mechanics specimen and may not be representative of cracking in service.

Results of corrosion fatigue crack growth rate tests for many metals have shown that the relationship between da/dN and ΔK can differ significantly from the three-stage relationship usually observed for tests in air, as shown in figure 2. The shape of the curve depends on the material/environment system and for some cases time-dependent (as distinct from cycle-dependent) cracking modes can ensue which can enhance crack growth producing frequency-dependent growth rate plateaux as shown in figure 2.

4.2 Specimens for corrosion fatigue crack propagation testing

4.2.1 General

A wide range of standard specimen geometries of the type used in fracture toughness testing may be used. The particular type of specimen selected will be dependent upon the form of the material to be tested and the conditions of test.

Pin-loaded specimens such as compact tension (CT) specimens are not suitable for tests with R values of zero or less than zero because of backlash effects. For such purposes four-point single edge notch bend (SENB4) or centre cracked tension (CCT) specimens loaded by friction grips are suitable.

A basic requirement is that the dimensions of the specimens be sufficient to maintain predominantly triaxial (plane strain) conditions in which plastic deformation is limited in the vicinity of the crack tip. Experience with fracture toughness testing has shown that for a valid K_{Ic} measurement a, B and $(W\text{-}a)$ should not be less than

$$2{,}5\left[\frac{K_{\mathrm{Ic}}}{\sigma_{\mathrm{y}}}\right]^2$$

where σ_{y} is the yield strength.

It is recommended that a similar criterion be used to ensure adequate constraint during corrosion fatigue crack growth testing where $K_{\max}$ is substituted for K_{Ic} in the above expression.

4.2.2 Specimen design

Specimen geometries which are frequently used for corrosion fatigue crack growth rate testing include the following:

a) three-point single edge notch bend (SENB3);

b) four-point single edge notch bend (SENB4);

c) compact tension (CT);

d) centre-cracked tension (CCT).

Details of standard specimen designs for each of these types of specimen are given in figures 3 to 6 and permitted notch geometries are given in figure 7. Suitable machining tolerances are given in table 1.

4.2.3 Stress intensity factor considerations

It can be shown, using elastic theory, that the stress intensity factor acting at the tip of a crack in specimens or structures of various geometries can be expressed by relationships of the form:

$$K_{\mathrm{I}} = Q\sigma\sqrt{a}$$

where

Q is the geometrical constant;

σ is the applied stress;

a is the crack length.

Stress intensity factors can be calculated by means of a dimensionless stress intensity coefficient, Y, related to crack length expressed in terms of a/W (where W is the width of the specimen) through a stress intensity factor function of the form:

$$K_I = \frac{YP}{BW^{1/2}}$$

NOTE — Where $P \leqslant 0$, $K = 0$. Nevertheless, it should not be assumed that negative loading will have no influence on the rate of crack growth.

The values of Y appropriate to the four specimen geometries discussed above are given in tables 2 to 5.

4.2.4 Specimen preparation

Specimens of the required orientation (see figure 1) shall, where possible, be machined in the fully heat-treated condition, i.e. in the material condition of interest. For specimens in material that cannot easily be completely machined in the fully heat-treated condition, the final heat-treatment may be given prior to the notching and finishing operations provided that at least 0,5 mm per face is removed from the thickness at the finish machining stage. However, heat treatments may be carried out on fully machined specimens in cases where heat treatment will not result in detrimental surface conditions, residual stresses, quench cracking or distortion.

After machining, the specimens shall be fully degreased in order to ensure that no contamination of the crack tip occurs during subsequent fatigue precracking or corrosion fatigue crack propagation testing. In cases where it is necessary to attach electrodes to the specimens by soldering or brazing for crack length monitoring purposes, the specimens should be degreased following this operation prior to precracking in order to remove traces of remnant flux.

4.2.5 Specimen identification

Specimen identification marks may be stamped or scribed on either the face of the specimen bearing the notch or the end faces parallel to the notch.

5 Apparatus

5.1 Environmental chamber

The environmental chamber shall completely enclose the test section of the specimen. Wherever possible, the gripped portions shall be excluded from contact with the solution environment to prevent galvanic effects and crevice corrosion. If this is not possible, appropriate measures shall be taken through, for example, the use of similar metals, electrical insulation or coatings. An adequate volume of solution to metal area ratio is required (dependent on reaction rates and exposure time) and a circulation system is usually necessary. For conditions of applied potential or applied current a separate compartment for the counter electrode may be necessary to limit any effects caused by reaction products from this electrode. Non-metallic materials are recommended for the environmental chamber and circulation system where this is practicable. These materials shall be inert. Note that glass and certain plastics are not inert at elevated temperatures. Where metallic chambers are necessary these shall be electrically insulated from the specimen to prevent galvanic interaction.

For tests in gaseous environment an all-metal-chamber is preferred.

5.2 Crack length measurement

The most commonly used techniques for the measurement of crack length are described in annex A. Optical methods of measurement are often precluded by the environment and test chamber and, in any case, provide guidance only to the surface length of a crack. Enhancement of crack visibility by removal of corrosion products may perturb the local electrochemistry and is not recommended. Methods that measure the average crack length across the thickness of the specimen are generally preferred. These include electrical resistance methods. AC and DC potential drop measurements are suitable but should be checked to ensure that they exert no detectable influence on the rate of corrosion fatigue crack propagation and appropriate methods should be used to eliminate galvanic effects. Compliance methods based on measurement of displacement across the notch or of strain in the back face of the specimen opposite the notch can also be used.

6 Fatigue precracking

6.1 General

The machine used for fatigue cracking should have a method of loading such that the stress distribution is symmetrical about the notch and the applied force should be known to an accuracy within ± 2,5 %.

In corrosion fatigue studies in the laboratory an artificial precracking procedure is introduced to provide a sharpened fatigue crack of adequate size and straightness. In principle, this procedure can affect subsequent crack growth depending on the frequency used, the manner in which the loading parameters are adjusted and whether precracking is conducted in air or in the test environment.

In some materials, the introduction of the corrosion fatigue test environment during the precracking operation will promote a change from the normal ductile transgranular mode of fatigue cracking to a less ductile corrosion fatigue mode. This may facilitate the subsequent initiation of cracking during corrsion fatigue testing. However, unless corrosion fatigue testing is conducted immediately following the precracking operation, corrodent remaining at the crack tip may promote blunting due to corrosive attack. For this reason, it is recommended that, unless agreed otherwise between the parties, fatigue precracking should be conducted in the normal laboratory air environment. In this case, precracking can be expedited by the use of high cyclic frequency.

6.2 Precracking procedure

Conduct fatigue precracking with the specimen fully heat-treated to the condition in which it is to be tested until the crack extends beyond the notch at the side surfaces by at least $0{,}025W$ or 1,25 mm, whichever is greater.

The final $K_{\max}$ during precracking shall not exceed the initial $K_{\max}$ for which test data are to be obtained. Ideally, precracking should be conducted without reduction in the value of $K_{\max}$. This is feasible for $\mathrm{d}a/\mathrm{d}N > 10^{-8}$ m/cycle but impractical for lower growth rates (see 6.3).

NOTE — The ΔK values to give growth rates of about 10^{-8} m/cycle are:

— steels, nickel, titanium and copper alloys: $\Delta K = 13\ \mathrm{MPa \cdot m^{1/2}}$

— aluminium alloys: $\Delta K = 6\ \mathrm{MPa \cdot m^{1/2}}$

The $K_{\max}$ value can be evaluated from the R value of interest. The value of $K_{\max}$ shall not exceed $0{,}7K_{\mathrm{Ic}}$.

The $K_{\min}$ value can be as important as $K_{\max}$ during precracking; $K_{\min}$ will dictate crack wake effects. For example, high R versus low R crack wake effects can dramatically affect corrosion fatigue testing results. Transient $\mathrm{d}a/\mathrm{d}N$ (crack closure influenced) behaviour can result.

At the end of precracking check that the surface crack lengths do not differ by more than $0{,}1a$. If the fatigue crack departs more than ± 5° from the plane of symmetry the specimen is not suitable for further testing.

The precracked specimen may be stored in a dessicated vessel until required. Long storage periods should be avoided because of possible crack tip blunting or contamination effects.

6.3 Precracking for low crack growth rates or ΔK_{th} determination

For $\mathrm{d}a/\mathrm{d}N < 10^{-8}$ m/cycle and for determination of the threshold ΔK (see 8.5) the precracking procedure described in 6.2 should be followed initially. A load-shedding procedure is then adopted until the lowest ΔK or crack growth rate of interest is achieved.

Cyclically load the specimen, smoothly varying K_{max} with crack length according to:

$$K_{\mathrm{max}} = K_{\mathrm{s}} \exp\left[C_{\mathrm{k}}\left(a - a_{\mathrm{s}}\right)\right]$$

where

a_{s} is the crack length after the preliminary precracking stage (see 6.2);

K_{s} is the corresponding value of K_{max};

C_{k} is a load shedding factor; ($C_{\mathrm{k}} = -100\ \mathrm{m}^{-1}$ is generally satisfactory when a and a_{s} are expressed in metres).

Continue load shedding, varying P_{min} so that the stress ratio R remains constant and equal to R_{s}, the value after the preliminary precracking.

NOTE — Continuous load shedding by computer control is recommended. If step shedding of load is employed the reduction in P shall not exceed 10 % of the previous value, and adjustments should not be made until the crack has grown by at least the prior plane strain plastic zone size $\left(R_{\mathrm{p}} = 0{,}1\left[K_{\mathrm{max}} / \sigma_y\right]^2, \mathrm{m}\right)$.

An alternative method of precracking for low crack growth rates or threshold ΔK determination can be used for high R values simply by increasing K_{min} while maintaining K_{max} constant until the relevant ΔK value is obtained.

Assuming the notch to behave as a crack of the equivalent length, cyclically load the specimen such that K_{max} equals the value of interest and K_{min} is derived from the target value of R.

When a reaches a_{s}, cyclically load the specimen, smoothly varying K_{min} with crack length according to

$$K_{\mathrm{min}} = K_{\mathrm{s}}\left[1 - \left(1 - R_{\mathrm{s}}\right)\exp\left\{C_{\mathrm{k}}\left(a - a_{\mathrm{s}}\right)\right\}\right]$$

where C_{k} is a load shedding factor ($C_{\mathrm{k}} = -280\ \mathrm{m}^{-1}$ is generally satisfactory when a and a_{s} are expressed in metres).

Vary P_{max} so that K_{max} remains constant and equal to K_{s}. Continue until the appropriate ΔK value is obtained.

NOTE — $K_{\mathrm{s}}\ (1 - R_{\mathrm{s}})$ which equals ΔK at the beginning of the determination can conceivably be less than ΔK_{th} at this value of $R = R_{\mathrm{s}}$ and this test method would clearly be inappropriate.

7 Test conditions

7.1 Environmental considerations

Because of the specificity of metal-environment interactions, it is essential that corrosion fatigue crack propagation tests are conducted under environmental conditions which are closely controlled (see paragraphs 3 and 4 below).

The environmental testing conditions depend upon the intent of the test but, ideally, should be the same as those prevailing for the intended use of the alloy or comparable to the anticipated service condition.

Environmental factors of importance are electrode potential, temperature, solution composition, pH, concentration of dissolved gases, flowrate and pressure. ISO 7539-1 provides useful background information. In relation to gaseous environments a critical factor is purity of the gas.

Tests may be conducted under open circuit conditions in which the electrode potential of the metal is dependent on the specific environmental conditions of the test, of which the degree of aeration is an important factor. Alternatively, the electrode potential may be displaced from the open circuit value by potentiostatic or galvanostatic methods.

Auxiliary electrodes to apply external current should be designed to produce uniform current distribution on the specimen, i.e the electrode potential should be constant.

7.2 Stressing considerations

7.2.1 Cyclic frequency

As in cycles to failure testing, cyclic frequency is usually the most important variable that influences corrosion fatigue crack propagation.

The rate of corrosion fatigue crack propagation generally increases with decreasing frequency because of the time dependence of the corrosion and diffusion processes that contribute to the corrosion fatigue process. At higher cyclic frequencies (generally greater than 10 Hz), the rate of corrosion fatigue crack propagation may be no greater than that of fatigue crack growth in air because insufficient time is available during each loading cycle for significant effects to occur. In some cases, the rate of corrosion fatigue crack propagation may also fall at very low cyclic frequencies because repassivation may outpace the rate of rupture of protective surface films at the crack tip.

Since too high or too low a cyclic frequency can lead to non-conservative data, it is important that corrosion fatigue crack propagation tests be conducted at a cyclic frequency that is relevant to the application under consideration. It is desirable to run tests at several frequencies both at greater and less than the application under consideration to assess the effects of changing frequencies.

7.2.2 Stress ratio

The rates of corrsion fatigue crack propagation are usually increased by higher stress ratios for several reasons depending on the system and including effects of stress ratio on crack tip straining, stress distribution ahead of the crack tip, crack tip shielding and crack chemistry. For this reason, the stress ratio used shall be representative of that encountered.

7.2.3 Waveform

For a given cyclic frequency, the waveform of the loading cycle governs the rate of film rupture at the crack tip and may, therefore, influence the rate of corrosion fatigue crack propagation. Hold periods at minimum, intermediate or maximum load in the cycle can either increase or decrease the rate of corrosion fatigue crack propagation, depending on the mechanism of the cracking process. For example, where K_{max} exceeds the K value for time dependent modes, a hold time at maximum load may be expected to increase the rate of crack propagation. However, in materials which are resistant to cracking under static load, a hold time at maximum load may reduce the rate of crack propagation because of time dependent crack blunting due to corrosion or plasticity. Such effects necessitate the use of an appropriate waveform and hold times during corrosion fatigue crack propagation testing.

Some practical applications involve exposure to random loading cycles or to well-defined periodic changes in the cyclic loading conditions. While some insight into the influence of these fluctuations may be gained by the summation of the effects observed during a series of tests under different loading conditions, it is preferable to simulate the service conditions by computer control using block or random loading programs.

7.2.4 Crack tip shielding (closure) effects

Rough intergranular corrosion fatigue fracture surfaces, oxides or calcareous deposits on the fracture surfaces can cause premature crack surface contact at a stress intensity factor $K = K_{\text{closure}}$ during unloading. This reduces the effective crack tip driving force below the applied ΔK and can greatly reduce fatigue crack propagation rates. Under these circumstances an effective ΔK is appropriate, as follows:

$$\Delta K_{\text{eff}} = K_{\text{max}} - K_{\text{closure}}$$

This reinforces the need for environmental and loading conditions to be carefully controlled during crack progagation measurements so that beneficial crack closure effects relevant to service can be identified.