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Corrosion of metals and alloys — Measurement of environmentally assisted small crack growth rate

Corrosion des métaux et alliages — Mesurage de la vitesse de propagation des petites fissures assistée par l'environnement

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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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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.

Introduction

A core requirement for life prediction of structures and components for which environmentally assisted cracking is a potential failure mechanism is establishing a reliable methodology for quantifying the rate of damage development through the different stages of its evolution. For long cracks, standards for measuring environment assisted crack growth rates are well established, there are extensive data for key industrial sectors and there is a degree of confidence in their engineering application, the latter coupled with advanced monitoring and non-destructive inspection techniques. In service, cracks initiate predominantly at the surface (at corrosion pits, inclusions, physical defects) and the early development of those small cracks could represent a significant fraction of the life of a component.

NOTE There are exceptions to cracks initiating at the surface in relation to fatigue crack initiation at sub-surface inclusions or in hydrogen generating environments including hydrogen induced cracking at internal voids.

However, there have been no standards for environmental-assisted small crack growth that guide the measurement process; simply recognition that the growth rate can be different from the crack growth for long cracks, that the rate will be sensitive to the local electrochemistry, near-surface gradients in microstructure, mechanical properties and residual stress, as well as loading conditions.

Corrosion of metals and alloys — Measurement of environmentally assisted small crack growth rate

1 Scope

This document specifies a method for determining the growth rate of small surface cracks in an aqueous environment (including atmospheric exposure) based on measurement of the change in size of the crack with exposure time.

The methodology can be applied to stress corrosion and corrosion fatigue crack propagation.

It also describes the varied methodologies for the generation of crack precursors including accelerated generation of single pits.

Industries for whom this document is relevant include power generation (including nuclear), oil and gas, aerospace and automotive.

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

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

ISO 8044, *Corrosion of metals and alloys — Basic terms and definitions*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 7539-1, ISO 7539-6, ISO 8044 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
small crack
crack that is initially small in three dimensions (in particular, both length and depth) in comparison to a relevant microstructural scale, continuum mechanics scale or physical size scale

Note 1 to entry: The crack can also be defined as small in terms of differences in crack-tip electrochemistry between a small and long crack with the transition between being environment dependent.

3.2
short crack
though-thickness crack in a fracture mechanics specimen that is initially small in two dimensions but long in one (through thickness)

Note 1 to entry: The crack can also be defined as short in terms of differences in crack-tip electrochemistry between a short and long crack with the transition between being environment dependent.

4 Principle

To measure the evolution of the small crack size with time at high resolution it is essential that the measurement probes or observational instruments are located as close as possible to the crack generation site. For that reason, a crack precursor is introduced in a controlled way and to a defined depth, within the constraints of the precursor generation method. There are different techniques for introducing crack precursors, including controlled corrosion pitting, mechanical notching, focus ion beam (FIB) milling, laser ablation and electric discharge machining. Very small notches may be desirable to locate the crack initiation site to a particular feature of the microstructure and here techniques such as FIB milling or laser ablation might be preferred.

NOTE Each of these techniques will affect the local microstructure and mechanical properties surrounding the precursor.

For many service applications, corrosion pitting is a precursor to cracking and for those applications accelerated pitting of the specimen is the preferred approach, particularly when investigating the pit-to-crack transition and in characterizing the impact of the precursor on the early stages of crack development. A fundamental challenge is to define the mechanical driving force for the crack in the presence of a precursor when the crack is physically small or small compared to the microstructure.

5 Specimen type and preparation

5.1 The specimen design shall ensure that cracking occurs only at the crack precursor and that the mechanical driving force can be readily defined. A flat tensile specimen is an example of a suitable specimen type, see Figure 1. However, the dimensions of the specimen are not fixed and these can be modified to accommodate, for example, a larger crack precursor. The specimen may be gripped in the shoulder hydraulically or loaded through pin holes. A key requirement is to ensure that the radius of the fillet region is sufficiently large that the stress concentration does not lead to cracking in that region. Similarly, in using loading pins, the shoulder dimensions should be such as to reduce the likelihood of cracking at the holes. Deburring of specimen edges by light manual grinding with fine grinding paper is advisable to minimize crack initiation at the edge.

Dimensions in millimetre

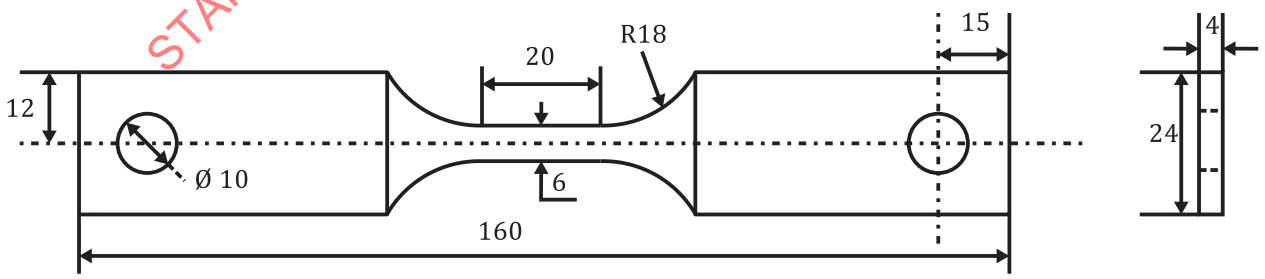


Figure 1 — Example of flat tensile specimen

5.2 The growth rate of cracks developed from small precursors will be particularly sensitive to the method of surface preparation. The method adopted shall reflect the intended service application, which may include as-processed, machined and ground, peened, or shall conform to a relevant test standard

for environment assisted cracking from plain surfaces. However, the rougher the surface the more challenging it can be to detect the early stages of crack development by optical methods. Prior to testing, a reference specimen should be examined to characterize near-surface gradients in microstructure (using for example electron backscatter diffraction), hardness and residual stress.

NOTE Grinding of some metals, such as austenitic stainless steels, can lead to generation of a nano-crystalline layer (the thickness of which will depend on the grit size and coolant, if used) and to significant near-surface work-hardening.

5.3 The specimen shall be degreased with an appropriate solvent following surface preparation.

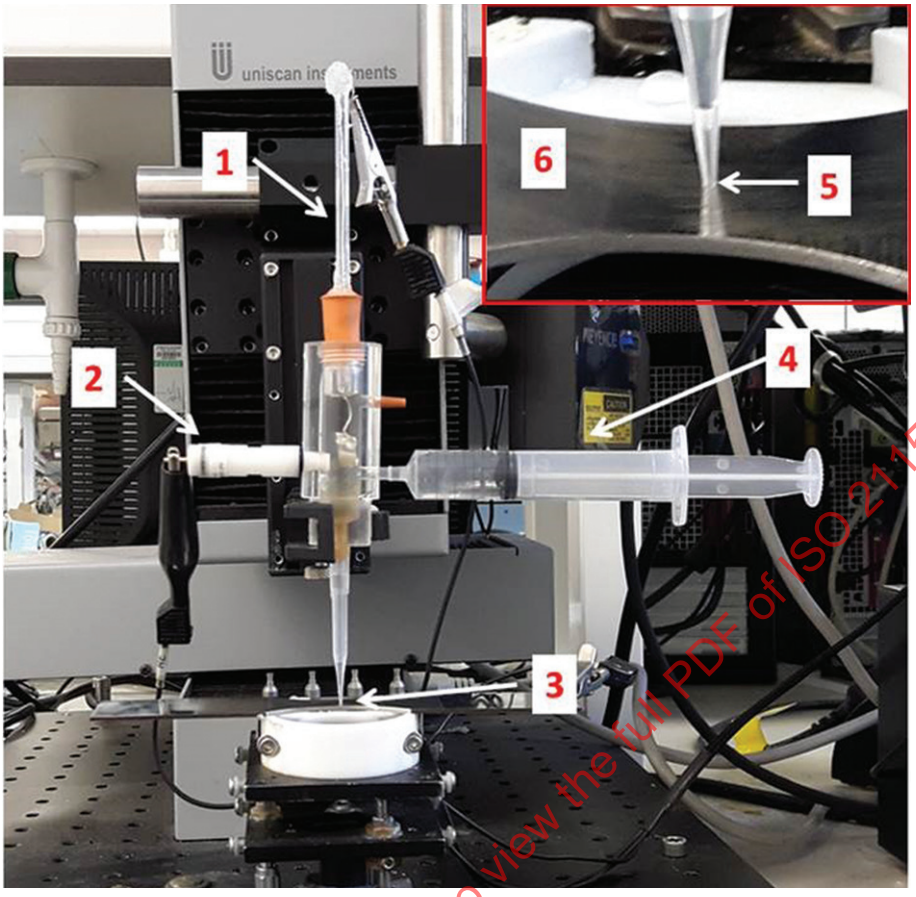
6 Crack precursor generation

6.1 Corrosion pits

6.1.1 The method of accelerated pit generation should be chosen such that a reasonably repeatable pit size can be generated with the pit geometry conforming as close as possible to that observed under natural corrosion conditions for the intended application. Methods adopted include partial coating of the specimen, and variations of the droplet technique. The technique adopted will depend on the metal-environment system.

6.1.2 For the coating method, a well-defined circular area of the specimen is left uncoated to limit the pit mouth diameter. Commonly, the specimen is then immersed in an aggressive environment appropriate to the metal for a period sufficient to generate a pit of the desired depth. Alternatively, an anodic current may be applied to the specimen (usually under galvanostatic control as that allows calculation of the volume of metal dissolved; from Faraday's Law). If the natural pit microtopography is important to the crack nucleating location care should be taken in selection of exposure time, solution and anodic current application to allow for a reasonably representative morphology to develop. In that context, a possible limitation of the technique is that undercut of the coating can occur and the pit geometry may not then be representative of a natural pit.

6.1.3 A related approach is to expose an uncoated specimen to an aggressive solution via a droplet fed from a small diameter capillary and allow the specimen to corrode. Such an approach has proven useful for developing pits in carbon steel and is exemplified in Figure 2. The method can be combined with anodic polarization of the specimen.



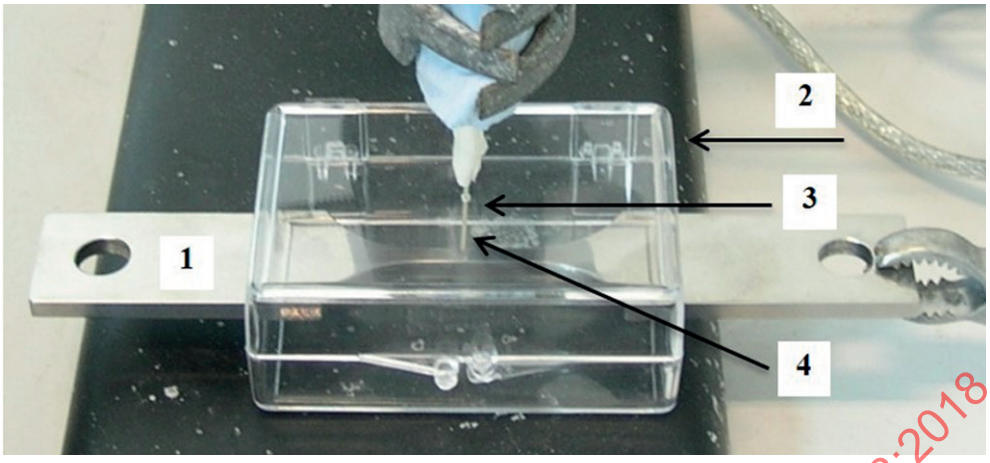
Key

1	auxiliary electrode	4	test solution reservoir
2	reference electrode	5	capillary tip
3	capillary tip	6	test sample

NOTE Inset: metal-electrolyte-capillary interface.

Figure 2 — Microcapillary cell for developing pit precursor as used, for example, by Akid et al.[1]

6.1.4 For corrosion resistant alloys, a similar type of droplet technique has been successful in terms of controlling pit size and geometry and involves applying a constant anodic current to the metal exposed to the droplet. The set-up for the latter is illustrated in Figure 3. When an anodic current greater than the passive current is applied to the electrode, the potential of the electrode will move in a positive direction until it reaches the pitting potential. A pit will initiate at the pitting potential and, as a result, the potential will drop rapidly to below the pitting potential, preventing additional pits from forming. Careful consideration is required in selection of the polarization current: if the applied current is too small, a pit may not initiate or the initiated pit can arrest as the pit area increases; if the applied current is too large, multiple pits can form and patchy corrosion can occur. Once the polarization current has been optimized, the desired depth of pit is achieved by controlling the polarization time. The virtue of the applied constant current method is that the method tends to give pit geometries that are macroscopically similar to those in service. Pitting is carried out usually with the aggressive anion similar to that in service. The depth of pitting can usually be controlled to a repeatability of about 10 % by a combination of applied current, solution conductivity and exposure time. However, the exposure conditions need to be explored for each metal-environment system in order to optimize the methodology and ensure that the artificially generated pit has the key features of real pits.



Key

1 specimen
2 cell for minimizing droplet evaluation
3 counter electrode (0,1 mm diameter Pt wire)
4 droplet (0,5 mm diameter)

Figure 3 — Droplet method of generating corrosion pit precursors using applied constant anodic current

EXAMPLE As an example of the galvanostatic droplet in relation to Figure 3, for a 12Cr martensitic stainless steel the volume of the droplet was 2 ml and the surface diameter of the droplet was less than 2 mm. The applied current was 20 µA and the sodium chloride concentration was 0,1 M. Polarization under these conditions for 300 s and 7 200 s generated pits with depths of 50 µm and 150 µm, respectively, both with a variation in depth of less than 10 % of the target value. The pits generated by this method give the truncated spheroidal geometry typical of pitting in stainless steel with a surface diameter approximately the same as the pit depth.

6.1.5 Pits generated by the different methodologies can become non-propagating in subsequent environmentally assisted crack growth testing, but this depends on the metal and alloy combination. Should pit growth occur during the test the increased cross-sectional area of the pit could impinge on the reliability of crack monitoring methods based on electrical resistance. The influence will be constrained predominately to crack depths smaller than the pit depth. When the crack depth is greater than the pit depth the effect should be minimal since the cross-sectional area is then determined only by the crack, provided that the crack growth rate continues to exceed the pit growth rate.

6.2 Mechanical notching

The most common approach to mechanical notching for small crack growth rate measurement is to drill a small hole to a specified depth. It is important to recognize that the method inherently introduces mechanical damage into the material that will influence the very early stages of crack development. Deburring the hole after drilling or reaming is essential since the burr can contain tiny cracks. Great care should be taken to retain the profile of the edge of the hole without any rounding-off, and to achieve a consistent finish within the hole by maintaining the sharpness of the tool and its feed rate and speed, but paying particular attention to maintaining tool condition.

6.3 FIB milling

FIB machining is typically undertaken with a gallium ion beam and simply erodes away the metal to the specified depth. The virtue is being able to generate very small notches (depths up to 20 µm) in specific

locations relative to the microstructure. The trench created in this manner should preferentially be orientated perpendicular to the stress axis unless specifically examining the effect of defect orientation.

NOTE 1 In some alloys, large inclusions can be present and these can be the site of crack initiation rather than the FIB notch.

NOTE 2 FIB with gallium ion beam will lead to some gallium ion implantation that could conceivably affect the early stages of corrosion from the notch region. Also, for stainless steels some local phase transformation has been observed.

6.4 Laser ablation

Laser ablation using nanosecond pulses can be used to generate pits/notches of 20 µm to 50 µm in depth, but with a small heat affected zone of a few micrometres. The heat affected zone can be eliminated using femtosecond laser beams with ultrashort laser pulses of high energy density. Since the pulse duration is extremely short no heat affected zone is formed at the edge of the ablation zone. A notch depth of about 50 µm is typical. Pit/notch mouth opening is of the order of 100 µm for these methods.

6.5 Electric discharge machining

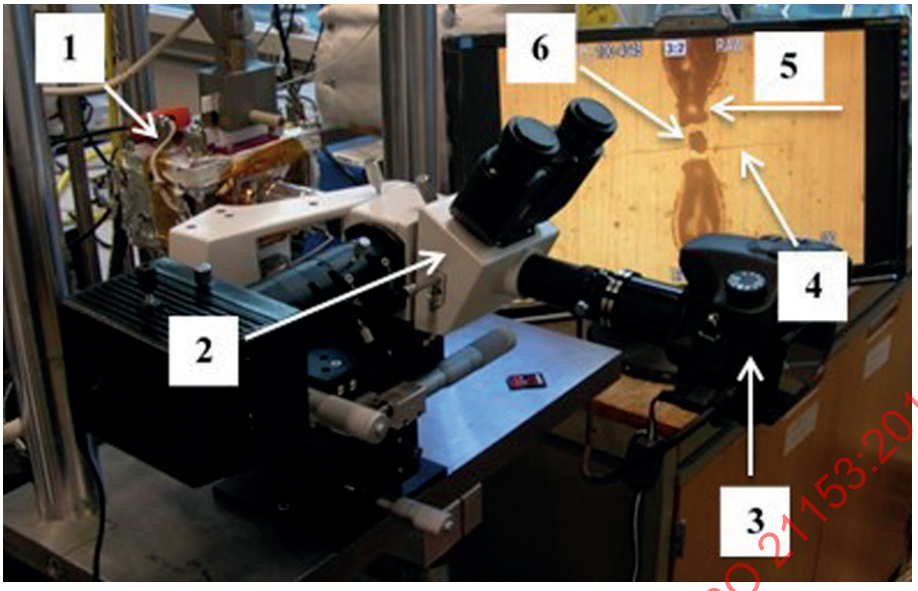
Electric discharge machining with fine wires (e.g. 0,2 mm diameter) can be used to generate small holes or notches, but inevitably the microstructure and mechanical properties of the material immediately adjacent to the hole/notch are modified. Copper deposition can occur from the brass wire often used, and some hydrogen uptake can occur. Hydrogen can be removed at elevated temperature with the choice of temperature chosen carefully to optimize the rate of removal without inducing microstructural change. Retention of deposited copper could induce a local galvanic effect.

7 Crack-size monitoring

7.1 Optical

7.1.1 For high resolution optical measurement of surface crack length, a combination of optical microscope and camera (photomicroscopy) is commonly adopted, as illustrated in Figure 4.

A high resolution digital camera with a fixed mirror to reduce vibration while capturing images is recommended. The camera is connected to an optical microscope with a long stand-off distance lens. The combination results in a theoretical resolution of about 0,07 µm per pixel with a lens of magnification 20 × , or 0,14 µm per pixel with a lens of magnification 10 × . However, the real spatial resolution would be about 0,3 µm, which is at the optical diffraction limit. The camera can be controlled with a signal from the testing machine control system and for low frequency loading synchronized with the fatigue load waveform to enable the image to be taken automatically at the maximum and minimum load during fatigue tests. However, at high frequency the vibration will limit optical resolution. Suitable fixings and an X, Y, Z stage can be used to move the camera into the optimum position and allow easy focusing and changing of the field of view.



Key

1	test cell	4	crack
2	microscope	5	probe
3	camera	6	pit

Figure 4 — Example of optical surface crack monitoring system showing also the probes on either side of the pit for monitoring potential drop

7.1.2 The inherent limitation of optical techniques arises when significant corrosion occurs on the sample surface, thus, the technique is often constrained to corrosion resistant alloys or testing in air. However, testing in solution can be limited even for corrosion resistant alloys. Corrosion product effluent from the crack itself could eventually obscure small cracks and make the optical method redundant. Hence, it is primarily useful in the early stages of crack development to link with electrical resistant methods or for reference tests in air.

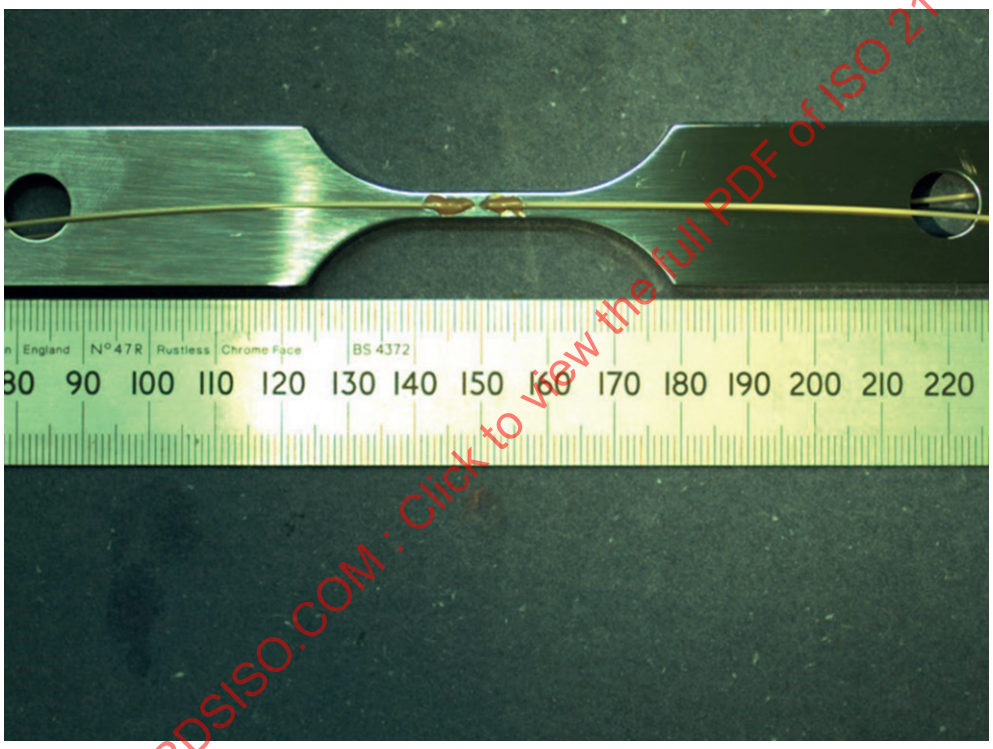
NOTE The same limitation of obscuration of the surface can limit application of techniques such as digital image correlation to detect the onset of surface cracking.

7.2 Electrical resistance techniques

7.2.1 The general use of direct current (DC) and alternating current (AC) potential drop (PD) methods for crack size measurement and possible sources of error are described in ISO 11782-2, with supplementary information included in Annex A. For small crack measurement, both techniques can be applied, although for long-term testing, the DC potential drop (DCPD) method tends to be the more stable; also, 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 instruments give very stable measurements. For AC potential drop (ACPD), the critical requirement 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. For the ACPD technique, the current flows through a thin layer below the surface of the crack (skin effect) and therefore "sees" a smaller effective cross-section. At low frequency it tends to the DCPD case, but at high frequency the depth of material round the crack that is sampled is smaller and there is greater sensitivity to crack size. However, for probes placed near the defect the advantage of the ACPD skin effect is minimal. In principle, multiple probes on either side of a crack mounted parallel to the crack direction

can, with analysis, give more insight into crack shape, with the centre pair primarily reflecting the depth of the crack, while the side pairs reflect crack symmetry and growth along the surface.

The primary issue is to ensure that the potential measurement probes are located as close to the crack precursor as possible (see, for example, Figure 5) without interfering with crack development in any way. In that context, the use of very fine wire (0,1 mm), such as platinum wire, spot-welded on either side of the crack precursor, is recommended. Insulating the electric wires from solution is recommended though it could be less necessary for corrosion resistant alloys in some environments as the surface area of the wire (which acts as the cathode) will be relatively small. However, if there is a possibility of localized attack at the joint this could undermine the spot welding. In this case, the platinum wire should be shielded from the environment. This can be achieved, for example, by insulating the wires with 0,4 mm polyetheretherketone (PEEK) tube and sealing with a stop-off lacquer or special heat resistant epoxy (with care to avoid spreading on to defect); the epoxy can also help to secure a stable support under fatigue loading. When working with alloys such as aluminium, copper wires may be preferred to platinum; however, joint damage size shall be accounted for, otherwise it could act as a nucleating feature rather than the intended defect.



NOTE The corrosion pit is not visible at this magnification, instead see Figure 4. The current leads are commonly attached at the ends of the specimen. In high resolution potential drop measurement there can be some sensitivity to the change in resistance due to the increase of plastic zone size with stress intensity factor.

Figure 5 — Example of test specimen showing attachment of DC potential drop probes (with PEEK insulation) on either side of corrosion pit

7.2.2 Shorting of the current across the crack walls can occur in some applications because of direct contact of the crack walls, bridging ligaments behind the crack, or conductive oxides. Some of these become apparent as cyclic or time-dependent variation of the potential drop with no crack growth. These effects tend to be greatest at lower values of the stress intensity factor where the crack opening displacement is small. An example is in fatigue testing as a consequence of crack closure and, where this has an impact, measurement of the potential drop should then be made at the maximum load where possible. In practice, synchronization of load and measurement is not always possible. To assess the impact, the effect of load on the potential drop can be investigated in the following way. Grow a crack and record the potential drop with no specific accounting for the position in the cycle. At set intervals, the fatigue test is interrupted and the load increased to the maximum value in the cycle. The potential

drop is recorded and the process repeated. Express the results in terms of V_c or V_c/V_0 with respect to whether the measurement was made during cycling or at the maximum load (V_c is the potential drop across the crack and V_0 is the initial value prior to crack initiation). A significant difference between the two methods of measurement will indicate that shorting between the crack walls is impacting the reliability of the measurement.

Where the various factors could be considered to impact on the crack size projected from the measured potential drop, the key requirement is to ensure that there is compatibility of the projected crack size derived with that determined by crack front marking; any deviation should be investigated in the context of possible sources of uncertainty outlined in this clause.

7.3 Calibration of crack measurement method

7.3.1 For both DCPD and ACPD techniques, a method of calibrating the measured potential drop shall be developed, preferably based on known crack size and crack profile measurements on a reference specimen. The primary unknown in the measurement process is the crack geometry.

7.3.2 For the potential drop method, calibration curves of crack depth (from crack front marking) versus potential drop are usually derived by fitting to the data with a suitable polynomial or the data are used to validate a theoretically based relationship (e.g. Reference [2]). The method of crack front marking adopted depends on the characteristics of the material and may include changing stress ratio in fatigue, changing frequency, heat tinting and changing environment exposure conditions in a systematic manner. The testing is usually undertaken using separate specimens with high frequency loading to accelerate data generation. However, it is conceivable that this may not induce the same crack geometry as that during the test (e.g. for stress corrosion cracks, which could be semi-elliptical with the long axis in the depth direction). Accordingly, the final crack geometry shall be examined to confirm the analysis. Ideally, the crack front should be at intermediate depths by undertaking interrupt tests at different exposure times. In all tests, the potential drop at the final measured crack length is used to benchmark the data and correct the test data, if necessary. If the shape of the observed crack front corresponds to that for the calibration, then the crack depth can be readily calculated assuming that same shape prevails during different stages of crack development. In the case of stress corrosion cracking, the potential drop calibration curve still responds to the area as a function of crack size, but the crack geometry can be different and this shall be accounted for when using analytical solutions, such as in Reference [2].

7.3.3 For optical measurements with no complementary potential drop measurements, the surface crack length is all that is known. To estimate the associated crack depth, the crack geometry adopted shall be based on assessment of the crack shape on the fracture surface post-test (see Clause 12).

8 Precracking

In some applications, the transition from crack precursor to a small crack may be accelerated by fatigue precracking at high frequency while crack monitoring (see ISO 11782-2[3] for further details of precracking). This can be desirable where only the kinetics of crack growth, rather than the transition phase, is of interest, or when testing at very low cyclic loading frequencies where the number of cycles for the transition to a small crack could be prohibitively long in terms of test duration. The unknown element in precracking from a corrosion pit is the crack depth. No technique can give this information as optical methods reflect only the surface length while electrical resistance techniques reflect the change in surface area or surface length of the crack (in both optical and electrical resistance techniques a crack shape would have to be assumed to estimate the depth). The actual crack shape and depth following precracking can sometimes be deduced by examination of the fracture surface after the test. The precracking may be conducted in air or in the test solution, provided in the latter case that the test is continued in solution without any pre-exposure to the atmosphere. Any exposure to air after precracking could lead to oxidation of corrosion products in the crack and also changes in corrosion potential that could impact on subsequent crack propagation.

9 Test apparatus

ISO 7539-1 should be consulted for general information on the apparatus and on control of the solution environment or atmospheric testing conditions as appropriate. Only the essential details are presented here.

The environmental chamber shall completely enclose the test section of the specimen. Wherever possible the gripped portions should be excluded from contact with the solution environment (immersion tests) to prevent galvanic effects or crevice corrosion. If this is not possible, appropriate methods to avoid such problems shall be taken, for example, through the use of ceramic pins, electrical insulation or coating. Non-metallic materials may be adopted for the test chamber and recirculation system where this is feasible. However, for testing under deaerated conditions permeability of oxygen through plastic components, such as tubing, will limit the extent of deaeration and flexible metallic tubing is recommended. The materials adopted shall be inert for the test temperature of interest. Note that glass and certain plastics may not be inert at elevated temperatures. Where metallic chambers are used, they shall be electrically insulated from the test specimen and shall exhibit a sufficiently low passive current density at the temperature of relevance such that the solution is not contaminated significantly with dissolution products. For testing under atmospheric conditions, the chamber should be such as to maintain controlled atmospheric conditions throughout the test.

10 Environmental considerations

The environmental testing conditions depend on 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 significance for tests in aqueous solutions are electrode potential, temperature, solution composition, pH, concentration of dissolved gases and flow rate (see ISO 7539-1). Tests may be conducted under open circuit conditions in which the electrode potential of the metal is dependent on the specific environmental condition of the test, or the electrode potential may be displaced from the open circuit value by potentiostatic or galvanostatic methods. For alternate immersion tests, the cyclic periods of immersion and drying and conditions prevailing during those periods should conform to the appropriate standard or be reflective of service exposure. When testing under atmospheric corrosion conditions only, the salt deposition method should be defined and the relative humidity, temperature and time variation of these parameters should be controlled and monitored.

NOTE Temperature fluctuations, controlled or uncontrolled, will affect the measured potential drop in electrical resistance techniques. Such changes can be accounted for by use of a reference specimen for parallel measurements in an uncracked material.

11 Test procedure

11.1 Undertake crack precursor development, if appropriate, according to the chosen method (see Clause 6) and remove any residue by suitable degreasing. For corrosion pits, ultrasonic cleaning in water and alcohol followed by drying in acetone or similar solvent is recommended to ensure residual anions are removed from the pit. Measure the dimensions of the notch or corrosion pit and for the latter in particular take a photographic image relative to a measurement scale so that the details of the local morphology at the surface are captured.

11.2 Where appropriate, fatigue precrack to the required surface length, as measured optically, with an applied maximum load less than that to be applied during the test such that the value of the maximum stress intensity factor (K) on completion is less than that for the subsequent fatigue or stress corrosion cracking test.

In testing specimens with retained residual stress, consideration shall be given to the possibility that these stresses can relax during machining and precracking.

11.3 Mount the specimen in the test cell and attach to the loading frame. Introduce the environment to the cell at the test temperature and commence loading on immersion of the crack precursor.

11.4 The environment shall be monitored and controlled during the test. For tests in aqueous solutions, the solution chemistry shall be maintained within the specified limits associated with the particular test standard by maintaining an adequate solution volume-to-metal area ratio (dependent on reaction kinetics and exposure time); a circulation system is usually necessary. In applications involving low conductivity water, periodic refreshment and/or use of an ion exchange column can be required to maintain the solution conductivity within the desired range. For applied potential or current, the system shall be designed to limit any effect of reaction products from the counter electrode. The temperature of the solution shall be controlled to ±2 °C, but tighter control is desirable to optimize the potential drop system. It is strongly recommended that the electrode potential be measured, using a reference electrode appropriate for the application. Care shall be taken to limit ohmic (IR) drop in the measurement of potential and to minimize contamination of the test solution from the reference electrode; for example, by using a double junction electrode.

11.5 The crack size parameter (surface length or potential drop) shall be recorded either continuously or at intervals of time or elapsed loading cycles (in fatigue) with sufficient discretization to enable high resolution crack growth rate determination.

11.6 Upon completion of the test, the specimen shall be removed from the cell, washed in water and alcohol, and rinsed with acetone or similar solvent. The specimens shall then be stored in conditions that avoid atmospheric corrosion; for example, using a desiccator cabinet.

11.7 The specimen shall then be fractured in air (e.g. by high frequency fatigue) and the final crack size (maximum depth and the surface length on either side of the precursor) measured to benchmark the electrical resistance measurements. The fracture surface should be photographed and non-uniformity in final crack geometry noted, as this could impinge on the determination of crack size with time. For those tests where precracking was undertaken, the fracture surface should be examined carefully to identify, where possible, the initial crack geometry and dimensions. The depth and dimensions of the crack precursor should also be recorded. For some systems, chemical cleaning of the specimen may be necessary to remove oxide product from the fracture surface. ISO 8407[4] provides a range of possible chemical cleaning methods for different metals. Validation of the cleaning method should be undertaken with a reference specimen before applying to the fracture surface and for the latter only one fracture surface should be cleaned initially.

12 Data analysis

12.1 Estimating the crack depth

12.1.1 General

12.1.1.1 The first stage is to convert the measurement parameter to a crack depth[2]. There is no ideal method for small cracks because no measurement technique gives a direct measure of depth. All methods of analysis are based on an assumed geometry, which can change during the course of the testing. In the case of fatigue, semi-elliptical cracks often tend towards a semi-circular shape as the crack grows. However, for stress corrosion cracks the cracks tend to be semi-elliptical in shape with the depth increasing relative to the surface length ratio as the crack propagates, though there are exceptions.

NOTE 1 In some systems, such as aluminium alloys, crack front markers (“beach” marks) to indicate the crack front can be introduced by varying the loading frequency or stress ratio. These crack front markers can also be used to determine the growth rate post-test. However, this is only an option if it can be demonstrated that marking the crack front by this method has no impact on the subsequent crack growth rate.

NOTE 2 Techniques such as X-ray computed tomography have been used in some research applications to image the evolution of cracks in 3D, but they are often not suitable for conventional crack size monitoring and, also, specimen thickness is very limited because of reduced transmission with increasing thickness.

12.1.1.2 Ideally, a combination of surface length measurement up to the point of obscuration by corrosion product, potential drop measurement and fracture surface examination should be combined to provide the best assessment of crack geometry and the most informed basis for crack depth estimation.

There are three distinct situations:

— one in which there is a surface breaking crack that extends in depth to at least that of the precursor (12.2);

— a surface breaking crack that develops at or near the surface (12.1.3), but with a depth initially less than that of the precursor (e.g. from a corrosion pit);

— a crack that develops sub-surface at the base of the defect (12.1.4), detected by DCPD, for example, and then extends towards the surface where it is detected optically.

12.1.2 Surface breaking crack with depth at least that of the precursor

Assume the initial crack shape is semi-elliptical with a uniform crack front extending to the surface and the crack depth is at least that of the precursor (see Figure 6). The axes of the ellipse are the half surface crack length, c, and the crack depth, a, respectively. The ratio of a/c is not fixed and can change with time. Hence, the surface crack length, 2c, measured optically, is useful to increase confidence in the approach. The final crack surface length, 2c, and the crack depth measured on the fracture surface are used for validation. In some cases, where surface crack length measurement is not possible because of obscuration of corrosion products, the adoption of a constant semi-circular geometry (a = c) may be adopted provided that it is at least compatible with the final crack front upon fracture.

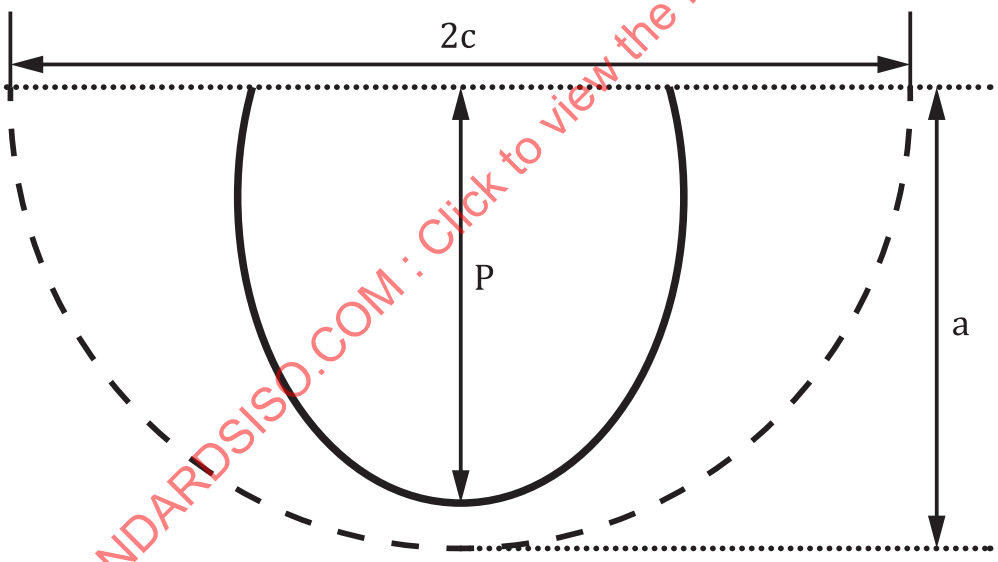
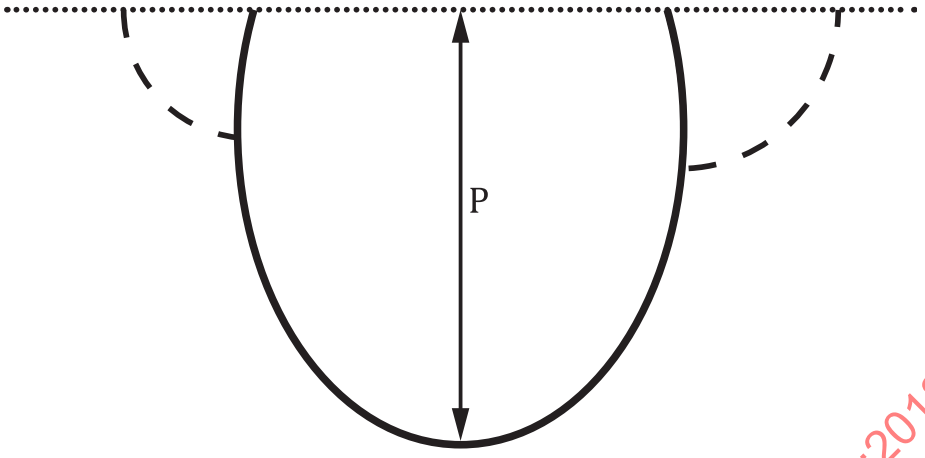


Figure 6 — Crack emerging from precursor pit or notch (P) with depth initially equal to that of the precursor

12.1.3 Surface breaking crack with depth less than that of the precursor

When the depth of the crack is less than the depth of the precursor (see Figure 7), which can be the case for cracks emerging from corrosion pits in some cases, and the cracks on either side of the pit have not yet merged to form a continuous crack front, defining the crack depth presents a challenge. In this case, the cracks are mechanically isolated and each crack has to be treated separately (a semi-circular crack shape is recommended as a default assumption) until such time as the cracks extend beyond the precursor depth and coalesce to form a continuous crack front (as in 12.1.2). In the latter case, a semi-circular crack shape may be assumed.



NOTE Crack depth is estimated based on an assumed semi-circular crack geometry as depicted in the figure.

Figure 7 — Cracks emerging near-surface on either side of the pit precursor with depth less than pit depth

12.1.4 Crack emerging at base of precursor but not surface-breaking

For situations where the crack emerges subsurface at the base of a pit or notch (see Figure 8) and is detected by the potential drop technique before breaking to the surface, the depth and shape of the crack can only be deduced by crack front marking.

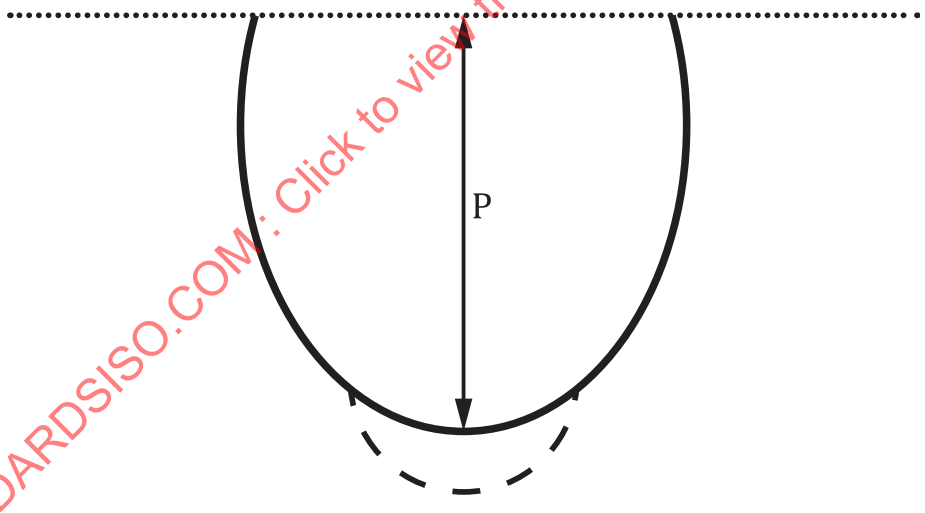


Figure 8 — Subsurface cracks initiated at the base of the pit

12.2 Calculating the small crack growth rate

12.2.1 The small crack growth rate is sometimes calculated based on the rate of change of surface length when using optical measurements, but more conventionally in aqueous environments based on the rate of change of estimated crack depth. There can be important differences in the trend. The small crack growth at the surface is often affected significantly by grain boundaries or other microstructural interfaces and this can give the appearance of growth that is slowing down as the crack reaches a microstructural "barrier" and then speeding up again. Indeed careful examination of high resolution photographic images readily supports this perception. However, interaction with the microstructure will be variable along the crack front so some averaging/smoothing of the overall crack growth process occurs. This smoothing is often reflected in a more damped fluctuation of crack growth rate when the growth rate is calculated based on potential drop techniques that reflects average position of the whole

crack front. Hence, calculating and interpreting the growth rate based on only surface measurement of crack length can be misleading and the focus should be on determination of the average crack growth rate along the crack front. The method of calculating crack growth rates has to distinguish measurement noise from intrinsic variability associated with fluctuation in growth rate due to interaction of the crack with the microstructure.

In long-term tests of material-environment systems exhibiting slow crack growth rates, it is not necessary to monitor at very small time intervals. Nevertheless, a minimum interval between successive crack size measurements is desirable to study the effect of crack size for small cracks with sufficient resolution, ideally much less than that of the material microstructure. This minimum interval relates to the extent of noise in the PD measurement and estimates of crack size. As an example, in corrosion fatigue, the cyclic crack growth rate, da/dN, can be significantly affected by measurement errors (see ASTM E647-13[5]). A direct-secant calculation of the average crack growth rate between two successive values of the crack depth measurements (a_1 and a_2) is exemplified for cyclic crack growth by Formula (1):

$$\frac{\mathrm{d}\tilde{a}}{\mathrm{d}N}=\frac{(a_2+\varepsilon_2)-(a_1+\varepsilon_1)}{\Delta N}=\frac{\Delta a}{\Delta N}+\frac{\Delta \varepsilon}{\Delta N} \tag{1}$$

where

a_2 and a_1 are two successive crack size measurements;

ε_2 and ε_1 are measurement errors.

Thus, as $\Delta a/\Delta N$ becomes very small, the error term $\Delta\varepsilon/\Delta N$ dominates the calculated value of $\Delta\tilde{a}/\Delta N$. Since small-crack data are often acquired at low crack growth rates, the crack extension between successive measurements tends to be small, and the growth rate data can exhibit an unusually large variability due to measurement error. Thus, in calculating the crack growth rate the value of the successive crack size increments should be based on an acceptable level of uncertainty. For example, for a measurement uncertainty of 0,3 µm in crack depth a crack size increment of 6 µm should be adopted to ensure an uncertainty in estimated crack growth rate of less than 5 %. Clearly, if the scale of the microstructure is less than that of the crack size increment some smoothing of the microstructural effect on growth rate would be inevitable.

NOTE Where crack front marking is possible (see 12.1.1.2), more discrete information on local growth rates at different positions along the crack front can be achieved.

13 Mechanical driving force

13.1 Comparison of the small crack growth rates with those obtained for long cracks requires the adoption of a consistent description of the mechanical driving force that can be applied at different length scales. This is commonly undertaken by application of linear elastic fracture mechanics (LEFM) and the stress intensity factor, K, for small scale yielding, but it is well established that LEFM cannot be applied with rigour to cracks that are physically small compared to the microstructure. More detailed microstructural-based fracture mechanics has been proposed, but it is non-trivial and not readily accessible to engineers. Formula (2) is proposed as a pragmatic approach to defining a nominal K, based on finite element analysis, for surface semi-elliptical and semi-circular cracks that are fully developed; i.e. the crack is surface breaking with a continuous crack front and a depth at least the depth of the precursor (see Figure 6):

$$K=\frac{\sigma_{\mathrm{t}}F}{\sqrt{Q}}\sqrt{\pi a} \tag{2}$$

where

