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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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Foreword

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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

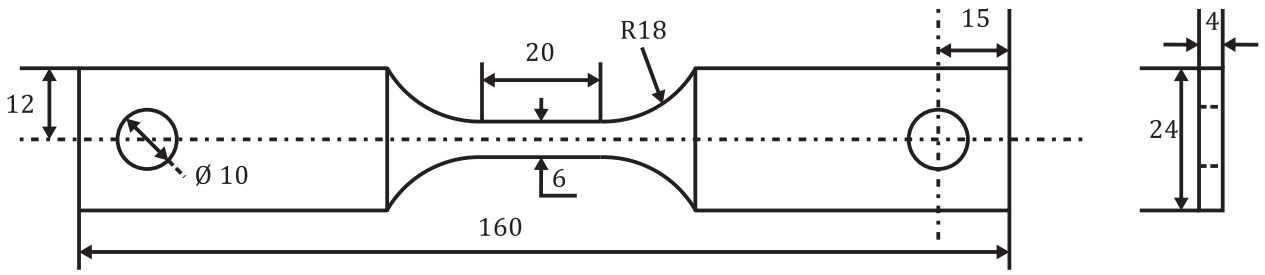


Figure 1 — Example of flat tensile specimen

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.

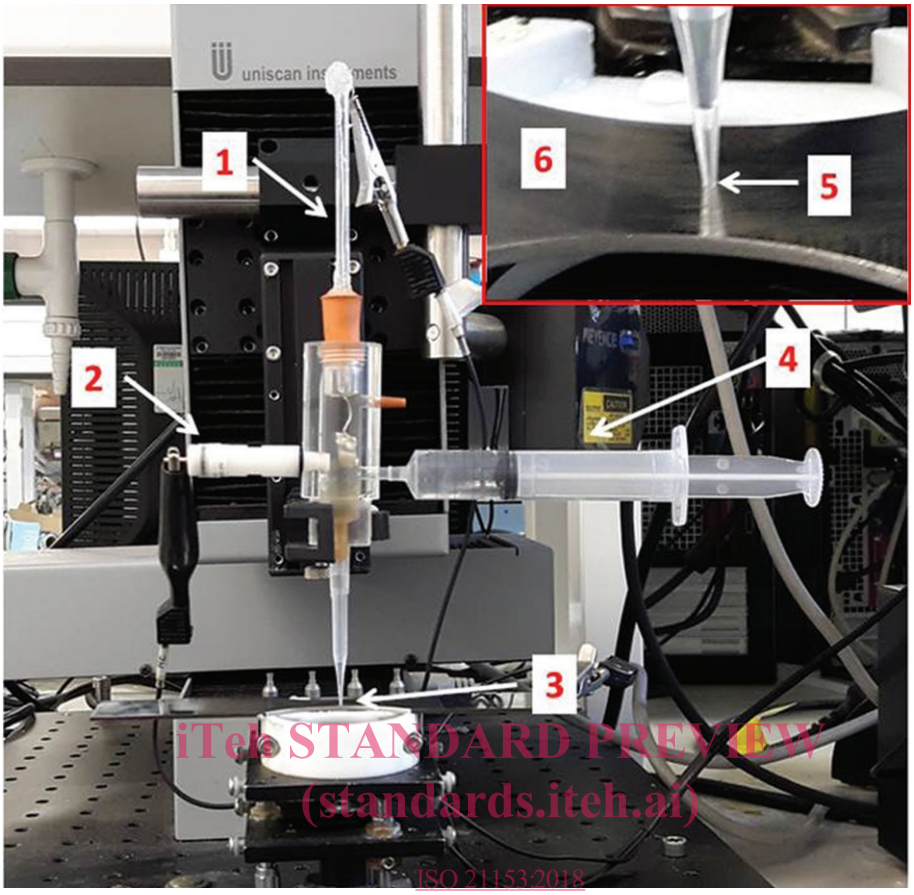
6 Crack precursor generation

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.



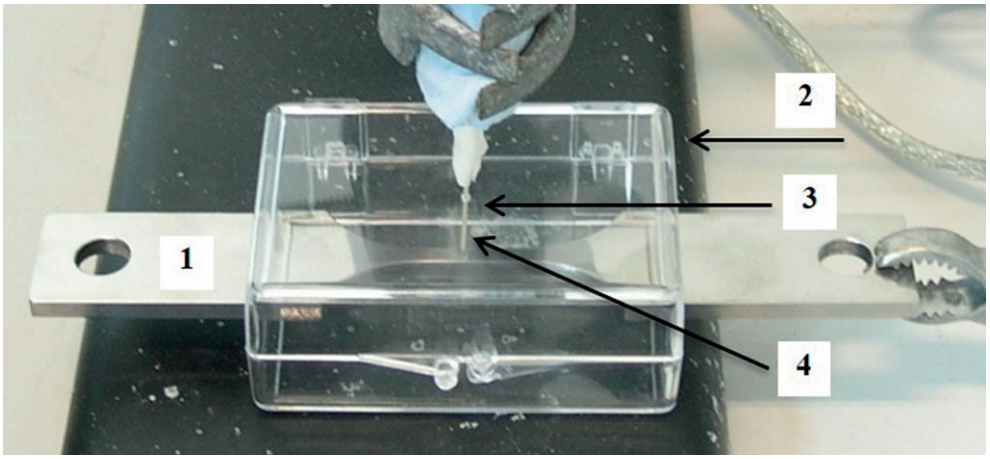
Key

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