INTERNATIONAL STANDARD

ISO 7539-1

Second edition
2012-12-15

Corrosion of metals and alloys — Stress corrosion testing —

Part 1:
General guidance on testing procedures

Corrosion des métaux et alliages — Essais de corrosion sous contrainte —

Partie 1: Lignes directrices générales relatives aux méthodes d'essai

Reference number
ISO 7539-1:2012(E)

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Published in Switzerland

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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.

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The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 7539-1 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

This second edition cancels and replaces the first edition (ISO 7539-1:1987), which has been technically revised.

ISO 7539 consists of the following parts, under the general title *Corrosion of metals and alloys — Stress corrosion testing*:

- *Part 1: General guidance on testing procedures*
- *Part 2: Preparation and use of bent-beam specimens*
- *Part 3: Preparation and use of U-bend specimens*
- *Part 4: Preparation and use of uniaxially loaded tension specimens*
- *Part 5: Preparation and use of C-ring specimens*
- *Part 6: Preparation and use of pre-cracked specimens for tests under constant load or constant displacement*
- *Part 7: Method for slow strain rate testing*
- *Part 8: Preparation and use of specimens to evaluate weldments*
- *Part 9: Preparation and use of pre-cracked specimens for tests under rising load or rising displacement*
- *Part 10: Reverse U-bend method*
- *Part 11: Guidelines for testing the resistance of metals and alloys to hydrogen embrittlement and hydrogen-assisted cracking*

INTERNATIONAL STANDARD ISO 7539-1:2012(E)

Corrosion of metals and alloys — Stress corrosion testing —

Part 1:
General guidance on testing procedures

1 Scope

1.1 This part of ISO 7539 describes the general considerations that apply when designing and conducting tests to assess susceptibility of metals to stress corrosion.

1.2 This part of ISO 7539 also gives some general guidance on the selection of test methods.

NOTE 1 Particular methods of test are not treated in detail in this part of ISO 7539. These are described in the additional parts of ISO 7539.

NOTE 2 This part of ISO 7539 is applicable to cathodic protection conditions.

2 Terms and definitions

2.1
stress corrosion
process involving conjoint corrosion and straining of the metal due to applied or residual stress

2.2
threshold stress
⟨stress corrosion⟩ stress above which stress corrosion cracks initiate and grow, for the specified test conditions

2.3
threshold stress intensity factor
K_{ISCC}
⟨stress corrosion cracking⟩ stress intensity factor above which stress corrosion crack propagation is sustained

Note 1 to entry: The threshold stress intensity factor is a concept of linear elastic fracture mechanics (LEFM) and is applicable when the plastic zone size is large compared with the microstructure and a high constraint to plastic deformation prevails; i.e. under plain strain-predominant conditions. For growing stress corrosion cracks, LEFM is not necessarily applicable in detail but is adopted as a pragmatic tool that is commonly used.

Note 2 to entry: Stress corrosion cracks may initiate at a surface or a surface defect and grow in the “short crack” regime at stress levels below the apparent threshold stress intensity factor. However, LEFM is not applicable in the short crack regime and sustained propagation of these cracks requires that the threshold stress intensity factor be exceeded.

2.4
test environment
either a service environment, or an environment produced in the laboratory, to which the test specimen is exposed and which is maintained constant or varied in an agreed manner

Note 1 to entry: In the case of stress corrosion, the environment is often quite specific (see Clause 6).

2.5
start of test
time when the stress is applied or when the specimen is exposed to the test environment, whichever occurs later

2.6
crack initiation time
period from the start of a test to the time when a crack is detectable by the means employed

2.7
time to failure
period elapsing between the start of a test and the occurrence of failure, the criterion of failure being the first appearance of cracking or the total separation of the test piece, or some agreed intermediate condition

2.8
slow strain rate test
test for evaluating the susceptibility of a metal to stress corrosion cracking that most commonly involves pulling a tensile specimen to failure in a representative environment at a constant displacement rate, the displacement rate being chosen to generate nominal strain rates usually in the range 10^{-5} s^{-1} to 10^{-8} s^{-1}

Note 1 to entry: Slow strain rate testing may also be applied to specimens in bend.

2.9
strain to failure
strain at which failure occurs in a slow strain rate test expressed usually as the plastic strain to failure See ISO 7539-7.

2.10
average crack velocity
maximum depth of crack(s) due to stress corrosion, divided by the test time

2.11
orientation
direction of applied tensile stress of a test specimen with respect to some specified direction in the product from which it was prepared, e.g. the rolling direction in the plate

3 Background

3.1 Although it is generally agreed that cracking is the usual result of stress corrosion, other manifestations such as intergranular corrosion or elongated fissures, which are enhanced by the presence of stress, have also to be recognized.

As far as this part of ISO 7539 is concerned, all phenomena involving metal dissolution or the action of hydrogen introduced into the metal as the result of simultaneous effects of a corrosive environment and a tensile stress are included, except for embrittlement by liquid metal and exfoliation corrosion.

3.2 There exists a wide diversity of methods used for assessing the stress corrosion properties of metals. Each has its own particular advantage in certain situations.

3.3 Stress corrosion cracking depends on both the exposure conditions and the mechanical and microstructural characteristics of the material and susceptibility or resistance to stress corrosion can only be defined in that context. Thus, for example, there is no intrinsic threshold stress intensity factor for a material.

3.4 Ideally, in order to establish the risk of stress corrosion in a given application, it is necessary to carry out simulation testing under all likely service exposure conditions. In practice, this is difficult, if not impossible, and rarely achieved, but a number of “standard tests” have been found as a result of experience to provide reasonable guidance on likely service behaviour for given specific applications. However, these laboratory “standard tests” are only appropriate to service conditions where experience has shown an appropriate relationship, however empirical, to exist. The fact that a given alloy passes or does not pass a test previously found useful in relation to another alloy may or may not be significant and a test that discriminates correctly between alloys used for a given application will not necessarily provide safe guidance if the exposure conditions are different. The use of a standard test beyond the point for which there is experience therefore requires validation.

3.5 In the following clauses, attention is drawn particularly to the fact that the stress corrosion process can be extremely sensitive to small changes in exposure or test conditions. The user of materials is responsible for selecting the conditions under which stress corrosion tests are performed and the fact that some tests are described in this part of ISO 7539 does not imply that these tests are the most appropriate ones for any given situation. The justification for describing these tests in a standard is that they are in widespread use and have been proven as valid for specific or common equipment-environment systems. However, the responsibility for interpretation of the test results remains with the user of materials and it is in no way diminished by the existence of this standard.

3.6 In addition to specific parts of ISO 7539 to cover the most widely used methods, it is considered that this more general document, concerned with the selection of test details and the interpretation of results, is required.

4 Selection of test method

4.1 Before embarking on a programme of stress corrosion testing, a decision has to be made regarding which type of test is appropriate. Such a decision depends largely upon the purpose of the test and the information required. While some tests attempt to reproduce service conditions as closely as possible and are of value to the plant engineer, others may be designed to study a mechanistic aspect of failure. In the former, for example, restrictions of material, space, time, etc., may mean the use of a relatively simple test procedure whereas in other circumstances more sophisticated testing techniques may be essential. Thus, studies of crack propagation rates may involve the use of pre-cracked specimens, although these may be inappropriate when considering, for example, the effects of surface finish. Although a number of sophisticated techniques are available, the adoption of a simple test may prove of great value in some circumstances when more elaborate techniques cannot be used.

4.2 When selecting a test method of the pass/fail type, it is important to realize that this should not be so severe that it leads to the condemnation of a material that would prove adequate for a particular service condition, nor should it be so trifling as to encourage the use of a material in circumstances where rapid failure would ensue.

4.3 The aim of stress corrosion testing is usually to provide information more quickly than can be obtained from service experience, but at the same time predictive of service behaviour. Among the most common approaches employed to achieve this are the use of higher stress, slow continuous straining, pre-cracked specimens, higher concentration of species in test environment than in service environment, increased temperature, and electrochemical stimulation. It is important however, that these methods be controlled in such a way that the details of the failure mechanism are not changed.

4.4 If it is too difficult to reproduce the service conditions exactly, it may be useful to analyse the stress corrosion process in order to determine as far as possible the main factors operating at different stages. The stress corrosion test then selected may involve only one step of the corrosion mechanism.

4.5 A brief guide to the selection of test methods is included in Annex A.

5 Stressing systems

5.1 General

Methods of loading test pieces, whether initially plain (i.e. nominally free from notches or pre-cracks), notched or pre-cracked, can be conveniently grouped according to whether they involve

a) a constant total strain (see 5.2);

b) a constant load (see 5.3);

c) an applied slow strain rate (see 5.4).

In the case of pre-cracked specimens, threshold conditions are defined in terms of a stress intensity value K_{ISCC} and tests may also be conducted under constant stress intensity conditions. Knowledge of the limitations of the various methods is at least as important as the choice of method of stressing.

5.2 Constant total strain tests

5.2.1 These form by far the most popular type of test as a group, since bend tests in a variety of forms come into this category. Furthermore, they simulate the fabrication stresses that are frequently associated with service failures.

5.2.2 Material in sheet form is frequently tested by bending; plate material is tested under tension or as C-rings, with the latter also used for testing tubular products and other semi-finished products of round cross-section.

5.2.3 Bend tests have the attraction of employing simple, and therefore frequently cheap, specimens and restraining jigs The tests may involve deforming the specimen plastically into a U-shape or adopting 2-point, 3-point, or 4-point bend configurations with a nominal applied stress at or below yield. For materials with a discrete yield point, elastic theory can be used to calculate the stress when testing at applied stresses up to that stress level. More commonly, and especially for corrosion resistant alloys, a discrete yield point is not observed and it is necessary to attach strain gauges to the specimen and deflect the specimen to achieve the desired level of total strain (usually up to a maximum of the 0,2 % plastic strain)

5.2.4 Tubular material may be tested in the form of C-rings or O-rings, the former being stressed by partial opening or closing of the gap and the latter by forced insertion of a plug that is appropriately oversized for the bore. The C-ring has also been found to be particularly useful for testing thick product forms, e.g. aluminium alloys in the short transverse direction.

5.2.5 Constant total strain tensile tests are sometimes preferred to bend tests because the initial stress is more readily characterized and through–thickness gradients of stress have to be considered in bend specimens.

5.2.6 The restraining frame used for either bend or tensile tests should be sufficiently stiff that constant displacement is maintained throughout the test.

NOTE The stiffness of the stressing frame employed may also influence the time to failure of a specimen because of stress relaxation, quite apart from any effect that it may have upon the initial stress level.

5.2.7 The use of restraining frames may be avoided by employing internally stressed specimens containing residual stresses as the result of inhomogeneous deformation. The latter may be introduced by plastic bending, e.g. by producing a bulge in sheet or plate material, or by welding. However, such tests involve problems in systematic variation of the initial stress, which usually achieves maximum values in the region of the yield stress. Moreover, elastic spring-back, in introducing residual stresses by bulging plate or partially flattening tube, may cause problems. Where welding is involved the structural modifications may raise difficulties, unless the test is simulative of a practical situation.

5.2.8 Constant total strain specimens are sometimes loaded by being placed initially into conventional testing machines or similar devices and then, while being maintained in the strained condition, having a restraining frame attached. When the load applied by the testing machine is removed, the specimen remains stressed by virtue of the restraint imposed by the frame, the assumption being made that the strain in the specimen remains constant as the restraint is transferred from the testing machine to the frame. Strain gauging can be used to confirm that there is no stress relaxation in the specimen. When testing at elevated temperature, consideration should be given to the change of material properties with temperature.

5.2.9 Stress relaxation may occur because of creep of the material, specimen thinning, or because some of the displacement is taken up by opening of the crack/s formed.

NOTE 1 Creep relaxation is most significant at elevated temperatures but can be important at ambient temperature in some cases (e.g. duplex stainless steels). The extent of relaxation should be assessed before testing and consideration of the value of constant total strain testing made, recognizing also that dynamic plastic strain is an inherent feature during any transient creep process.

NOTE 2 Specimen thinning is best assessed at the end of the test and the increased effective stress evaluated, accounting for any significant non-uniformity of thinning.

NOTE 3 The extent to which crack opening relaxes the stress will be dependent on the number of cracks formed, which will be material-environment sensitive. In some cases, the relaxation can be such that the specimen does not fail. Thus, post-test inspection for the existence of cracks is always required, the presence of which will constitute a failure.

5.3 Constant load tests

5.3.1 These may simulate more closely stress corrosion failure from applied or working stresses. Also, since the mechanical driving force increases as a crack propagates, such tests are more likely to lead to early failure or total failure than are constant total strain tests (see 5.2).

5.3.2 The relatively massive machinery usually required for dead-weight loading tests upon specimens of appreciable cross-section is sometimes circumvented by the use of a compression spring. The spring characteristics are chosen to ensure that the relaxation that occurs during testing does not significantly change the load. In the same category are modified proving rings used in the calibration of tensile testing machines. The axial load applied to a tensile specimen contained within the ring can be determined from measurement of the change in diameter of the calibrated ring.

5.3.3 An alternative approach for minimizing the size of the loading system is to reduce the cross-section of the specimen, e.g. by the use of very fine wire. However, it is dangerous to reduce the cross-section too far unless failure by stress corrosion is confirmed by, for example, metallography. This is because, in some stress corrosion environments, failure may result from pitting or other forms of attack with an attendant increase in the effective stress to the ultimate tensile strength of the metal. Other dangers are attendant on the use of specimens of very small section (see 8.2.2).

5.3.4 The cost of testing specimens under constant load on individual testing machines can be minimised by testing chains of specimens on a single machine. This practice also reduces the test chamber requirements. Chains of uniaxial tensile specimens can be connected with simple loading links, but this approach is better suited to situations where failures are not anticipated since the failure of a single specimen would invalidate the remainder. Chains of more compliant pre-cracked specimens can be connected with loading links, which are designed to progressively unload specimens as crack growth occurs in order to avoid disturbance to the other specimens that would otherwise be inevitable in the event of a failure. Users of such chain-linked specimens have to demonstrate that failure of an individual specimen does not invalidate the testing requirements for the other specimens.

5.3.5 The use of a tension specimen having a tapered gauge length has the obvious attraction of providing a range of initial stresses in a single specimen. However, caution should be exercised with their use in, for example, determination of accurate threshold stress levels. The stress gradient is critical. Also such factors as number of cracks present and net section yielding may influence the result. It may be more appropriate to use such specimens in “screening” tests to be followed by more limited conventional testing.

5.3.6 Constant load tests involve an increasing stress situation as cracks propagate; therefore, cracks once initiated are less likely to stop propagating than in the case of constant total strain tests for which stress relaxation may occur.

5.4 Slow strain rate tests

5.4.1 The application of slow dynamic straining is an important adjunct to conventional constant total strain or constant load testing because dynamic plastic strain is a key factor in the process of crack initiation and propagation. Indeed, localized dynamic straining will play an important role in the failure process in constant total strain or constant load testing.

Nevertheless, while recognizing the mechanistic significance of the process, for engineering purposes, the conventional use of the method remains primarily as a sorting or screening test. With very few exceptions, there are no acceptance criteria based on slow strain rate testing and quantifying actual strain rates in service remains elusive.

The slow strain rate method is most commonly used for testing of plain tensile specimens but has also been adapted for pre-cracked fracture mechanics specimens (see ISO 7539-9 for the determination of the threshold stress intensity factor).

In essence, the method involves the application of a relatively slow strain or deflection rate (e.g. 10^{-6} s^{-1}) to a specimen, under the appropriate environmental influence, until failure occurs.

5.4.2 Early use of the test was in providing data whereby the effects of such variables as alloy composition and structure, or inhibitive additions to cracking environments, could be compared and also for promoting stress corrosion cracking in combinations of alloy and environment that could not be caused to fail in the laboratory under conditions of constant load or constant total strain. Thus, it constitutes a relatively severe type of test in the sense that it frequently promotes stress corrosion failure in the laboratory where other modes of stressing plain specimens do not promote cracking.

5.4.3 The equipment required for slow strain testing is simply a device that permits a selection of strain rates while being powerful enough to cope with the loads generated. Purpose-built apparatus usually consists of a moderately stiff frame and a drive mechanism through a series of reduction gears that allows a selection of crosshead speeds in the range 10^{-3} to 10^{-8} $mm \cdot s^{-1}$. Indeed, it is the crosshead rate that is commonly controlled to give the desired nominal strain rate.

Plain or pre-cracked specimens in tension may be used, but if the cross-section of these would need to be large or the loads high, bend specimens may be used.

NOTE In most common use in testing of plain tensile specimens, the displacement of the crosshead is measured rather than that of the specimen. Accordingly, the measured displacement includes displacement of all features in the load train including the load cell. Thus, the apparent elastic modulus does not relate to the specimen but will depend on the test system as a whole. Similarly, the actual elastic strain rate on the specimen will also vary from one test assembly to the other. However, when plastic deformation occurs most of the displacement then occurs in the specimen and test system sensitivity becomes less significant.

5.4.4 It is important to appreciate that the same strain rate does not produce the same cracking response in all systems and that the rate has to be chosen in relation to the particular system being studied. Thus, while a strain rate of 10^{-6} s^{-1} is often employed in screening tests, the absence of stress corrosion cracking at this strain rate does not preclude the possibility that stress corrosion failure may be induced at a lower strain rate, e.g. 10^{-8} s^{-1}.

6 Environmental aspects

6.1 General

Historically, stress corrosion cracking was considered to occur in rather specific alloy/environment combinations, e.g. austenitic stainless steels in chloride solutions, mild steels in nitrate solutions, brass in ammoniacal solution. However, the list of such combinations continues to grow with time and instances of the cracking of materials in a very wide range of environments including high-purity water have been observed. Nevertheless, it is important to recognize that relatively small variations in environment conditions are important, requiring close attention to detail in solution preparation and control of all environmental parameters during testing.

The three key features of environmental control are temperature, water chemistry and flow rate with applied electrode potential an additional factor where appropriate.

While this part of ISO 7539 is concerned primarily with aqueous environments, exposure to organic fluids and gaseous environments may also induce cracking. In the latter case, gas pressure and purity will be the key variables to control.

6.2 Temperature

The significant influence of temperature upon chemical processes is well known, with reaction rates generally increasing in response to a rise in temperature. While this may also be the case in many corrosion processes, the influence of temperature is, for a number of reasons, frequently more complex. An increase in temperature, together with a corresponding increase in reaction rates, may result in a reduced corrosion rate due, for example, to the more rapid formation of protective films. In other systems, an increase in temperature may be necessary to activate localized corrosion as a precursor to stress corrosion cracking. However, too high a temperature may enhance the localized corrosion rate to the extent that the transition to a crack does not occur. Similarly, the reduction in the solubility of oxygen in aqueous solutions, which may accompany an increase in temperature, can also have an effect, and other examples could be quoted. Thus, there may be temperature windows for cracking, some of which may be experienced only during system transients. These could include start-up and shut-down or system upsets (e.g. cooling water failure causing a transient rise in temperature)

In view of the foregoing, it is clear that the test temperature should be closely controlled and whenever possible this should be selected to correspond to that expected in service, including scheduled transients. Although, as indicated in 4.3, increased temperature is sometimes used to accelerate test results, clearly such an approach must be undertaken with caution.

6.3 Water chemistry

6.3.1 The water chemistry adopted should represent as closely as possible the intended service environment. Nevertheless, for screening purposes, standardized environments are often adopted. These should be chosen carefully to be representative of the primary features of the plant environment as the relative susceptibility of alloys can change with change in solution composition.

In relation to simulating service environments, it is important to remember that local concentrations may occur; e.g. in crevices or where heat transfer takes place across interfaces, and that the bulk environment may not be that which causes cracking. Hence, it may be necessary to simulate those characteristics in testing (e.g. creviced specimens).

6.3.2 In preparing the solution, the baseline water quality depends on the application, with distilled or deionised water being adequate for preparing artificial seawater for example but high-purity water of conductivity less than 0,1 μScm^{-1} being necessary for simulating boiling water reactor environments. It is also important to recognize that the chemicals used in synthesis should be at least of analytical reagent grade quality. It is prudent to check the assay of these and to consider a higher grade where the concentration of minor constituents might be significant, as when using concentrated solutions. This is a consideration also when testing in strongly acidic or alkaline solutions.