
**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

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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*

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 $10^{-8} \text{ mm}\cdot\text{s}^{-1}$. 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 $0,1 \mu\text{Scm}^{-1}$ 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.

6.3.3 There are situations where relatively small changes in environment can promote marked changes in cracking response. The possible problems associated with the use of nominally 42 % boiling $MgCl_2$ for testing stainless steels may serve as an example. Since the hydrate of $MgCl_2$ is hygroscopic, solution preparation by weighing may lead to appreciable differences in boiling point and hence in time to failure in a stress corrosion test; it is therefore preferable to prepare the solution by adding water to the hydrate to achieve a particular boiling point.

6.3.4 In all testing, the test solution should ideally remain constant for the duration of the test. For some standardized environments, some change is tolerated for pragmatic reasons, for simplicity of testing and cost. More generally, it is important to ensure that the solution pH and dissolved gas content remain within acceptable tolerances and that contamination of the solution with reaction products does not influence the stress corrosion cracking behaviour. Furthermore, solution contamination directly or indirectly from the test cell needs to be considered. Examples include dissolution of silicates from glass at elevated temperatures, release of additive from plastics, permeation of oxygen through plastic tubing in de-aerated systems.

Ideally, the electrode potential of the test specimen should be monitored (where practicable) as this will provide critical insight with respect to the state of the metal and reaction processes as well as providing an important index of system stability.

6.3.5 The effects of varying the pH of the environment in relation to general corrosion are well recognized and, where appropriate studies have been made, the effects upon stress corrosion are no less marked. Changes in the pH of an environment during a test may be as important as the initial pH. The pH change during a test depends upon the volume of solution and the surface area of the specimen exposed, as well as upon the duration of the test. Use of a relatively large volume of solution with a small exposed area of metal, or replenishment of solution during the test, is likely to be associated with smaller pH changes and hence possibly different times to failure than would be the case with a small solution volume and large exposed area; indeed, if these quantities are sufficiently small and large respectively, failure may not occur at all in some systems. If tests are carried out with anodic stimulation, these effects of pH change may be aggravated, especially if the counter electrode is immersed in the stress corrosion cell. Indeed, in some cases where electrochemical stimulation is used, solution decomposition may occur to such an extent that the mechanism of failure is significantly changed from that which occurs at the free corrosion potential. Buffered solutions are sometimes used to overcome these problems, but their use may alter the mechanism of cracking or even inhibit this mode of failure and should be considered carefully.

The extent of pH increase that can be tolerated will be system dependent, but an increase constrained to below 0,2 is advised.

It should also be recognized that a pH increase may occur at the test-solution interface but be less marked in the bulk solution. For this reason, stirring of the solution can be important though this should be considered in the context of the application. For some alloys, local solution chemistry changes at the surface may be a key factor initiating localized attack and the onset of stress corrosion cracking.

6.3.6 Where oxygen plays an important part in the corrosion reactions that promote cracking, small changes in oxygen concentration can play an influential role because of the effect on the corrosion potential and corrosion products. Thus, in the testing of certain aluminium alloys in aerated solutions, failure may occur in several hours, but in de-aerated solutions (lower corrosion potential) no cracking may result in extended tests

For some systems, e.g. low alloy steel in high-temperature pure water, the corrosion potential can change sharply (by as much as 600 mV) over a small oxygen concentration range. For that reason, measurement of oxygen concentration in some systems is critical.

Even at ambient temperature, it may be prudent to ensure maintenance of oxygen concentration by bubbling air through the solution or by agitating the air-solution interface.

When oxygen reduction is mass transport limited, stirring of the solution will have an important influence on the corrosion potential. Enhanced transport of oxygen to the metal surface occurs also during drying out of thin liquid layers as in tests involving spraying, dripping and alternate immersion.

6.3.7 Conductivity measurement should be made when testing solutions of initially low conductivity to ensure that this is maintained.

6.4 Flow rate

The flow rate of the test solution can affect several factors: the corrosion potential, the near-surface solution chemistry, the solution chemistry in a crack, and, on a more mundane level, maintenance of the bulk solution chemistry in a complex re-circulation loop. In well-stirred solutions, mixing ensures maintenance of the bulk chemistry at the metal surface whether in service or in the laboratory. The flow rate adopted in testing should reflect the intended service application, within the constraints of practical laboratory testing.

In quoting the flow rate, the linear flow rate (m s^{-1}) should be defined as well as the rate of refreshment of the test cell.

6.5 Electrochemical aspects

6.5.1 The electrochemical nature of the reactions involved in stress corrosion cracking permits the cracking to be influenced by the application of current or potential from an external source. In most laboratory testing, this is achieved under potentiostatic conditions for which the potential is controlled, although galvanostatic methods (current control) or sacrificial anodes have been adopted. The response to the potential change in relation to cracking will be system dependent. Often there are potential regions within which cracking occurs with different mechanisms of cracking prevailing. The reason for applying the potential is often to identify those regions and to assess the likelihood that these will be established in service, e.g. by varying oxidising conditions, cathodic protection or indeed temperature changes.

6.5.2 The effect of potential upon cracking varies from one system to another, but some aspects of this subject may conveniently be discussed in relation to the cracking of carbon steels. Some tests have shown that these materials fail in different ranges of potential according to whether they are immersed, for example, in hydroxide, carbonate or nitrate solutions. The free corrosion potential of this material, in the respective solutions, usually lies within the cracking range in the case of nitrate but outside the cracking range in the case of other solutions. This indicates that, under these particular test conditions, failure could occur in nitrate at the free corrosion potential, but this would not be so in the hydroxide or carbonate solutions. This does not mean that carbon steels would never fail by stress corrosion cracking in these latter two environments at the free corrosion potential, but simply that this particular steel in the particular solutions used in these experiments did not fail at the free corrosion potential. The latter is, of course, dependent upon the composition of the steel, its surface conditions, and the composition of the environment.

6.5.3 It is possible, therefore, that some small additions to the environment, added intentionally or present as impurities, may cause the corrosion potential to lie within the cracking range so that stress corrosion results without applied potential. This has been interpreted as the effect of small additions of lead salts to NaOH solutions, which are well known to promote caustic cracking in laboratory tests that, in the absence of the lead additions, do not produce cracking. Other small changes in the composition of steels may have a similar explanation, at least in part. Thus, the effect of small Al additions to carbon steel in increasing cracking resistance, and of Cu additions in decreasing resistance, may be due to the former resulting in a more negative and the latter in a more positive corrosion potential, with consequent effect upon the cracking propensity. These examples illustrate the possible effects of relatively small changes in potential, not exceeding about 100 mV, in producing very marked changes in cracking response. They point to the necessity, especially in laboratory tests attempting to simulate a service failure, of reproducing the environmental conditions, and especially the relevant potential with precision.

6.5.4 When it is established that stress corrosion occurs only over a critical range of potentials it is possible to monitor by online potential measurements whether cracking is likely to be occurring in service equipment. Moreover, in some circumstances the risk of stress corrosion can be reduced or completely avoided by maintaining potentials outside the critical range either by the introduction of "inhibitive" substances or by cathodic or anodic protection.

6.5.5 The use of a potentiostat in the laboratory tests, while apparently far removed from most service conditions and adding to the cost of testing, is frequently the most efficient way of achieving particular potentials, and probably gives the added advantage of better reproducibility of results. It is frequently the case that mechanistic studies require involvement in electrochemical tests, but for laboratory work aimed at studying service failures, tests at the free corrosion potential (provided that this is known for the service condition) are often more realistic. It is important to realize, however, that the free corrosion potential depends upon a number of factors such as surface condition, time of exposure, etc., and thus the value in a laboratory test using machined or polished surfaces may be appreciably different from those in a service situation involving, for example, mill-scaled or rusted surfaces, even in the same environment. The decision to apply electrochemical control in tests simulating a service failure for the purpose of reducing the time factor or to obtain better reproducibility is only justifiable when the conditions outlined above are fulfilled. Otherwise, a better approach, if the reproducibility of laboratory data are not considered adequate, is to conduct a properly designed and statistically significant set of experiments.

6.5.6 In view of the significant effect of potential upon stress corrosion behaviour, it is necessary to take precautions to insulate electrically test specimens from other metallic components in test equipment that is immersed in the test solution.

6.5.7 It is also important to remember that the potential at a crack tip, especially when pre-cracked specimens are used, may be different from that at the surface where the crack emerges and where the potential is usually measured. The potential changes along cracks may sometimes be quite small (a few millivolts) but in other situations may reach hundreds of millivolts.

7 Time dependent issues

7.1 General

Laboratory tests are inherently short compared to the lifetime of the component or structure but it is important to be aware of factors that can compromise the results if the test time is too short for crack initiation and leads to non-conservative prediction. Typical time-dependent processes of relevance to initiation include pitting corrosion, intergranular corrosion, hydrogen uptake and changes in the characteristics of surface films with exposure time.

7.2 Pitting and intergranular corrosion

The issue with localized corrosion arises because of the conflict between minimising exposure time, to reduce testing costs, while recognizing that the depth of attack will progressively increase with time, albeit at a continually decreasing rate. Where localized corrosion acts as a precursor to stress corrosion cracking, there may be no alternative but to conduct long exposure tests.

7.3 Hydrogen uptake

Where hydrogen embrittlement is the primary failure mechanism, e.g. cathodically-protected high-strength steels, a fundamental question is how long should the duration of a laboratory test be to ensure that hydrogen uptake is sufficient to reflect behaviour in service, for which exposure times are of the order of years.

The key issue is the distance between the site of cracking and the primary source of hydrogen atoms. If the latter is remote then test times need to reflect this or pre-exposure should be considered. To predict the primary source of hydrogen atoms, some insight into the electrochemistry of the system is necessary. The kinetics of hydrogen atom generation can vary between the crack tip and the surface external to the crack because of differences in local pH, concentration of other species (e.g. H₂S) and electrode potential. The significance of these factors depends on whether the alloys are in the active or passive state. In the latter, the crack tip tends to dominate because the passive film reduces hydrogen uptake markedly, whereas for alloys in the active state it is possible that hydrogen generated on the

surface external to the crack may be important. In such a case, diffusivity measurements can provide estimates of the time to charge up the specimen.

8 Specimen design and manufacture

8.1 General

8.1.1 Specimen size is one of the initial considerations and the eventual choice depends upon a number of factors and usually involves a compromise. The cost and availability of material in the relevant metallurgical condition may, on the one hand, restrict the size of specimen possible, as may limitation of test equipment (e.g. available loading, volume of test chamber, etc.). On the other hand, the use of larger specimens may have the advantage of being more representative of the bulk material and may also avoid the problems of general corrosion or pitting which could arise using specimens of small cross-section, e.g. very fine wires.

8.1.2 The orientation of test specimens taken from bulk material, in relation to shape and orientation of grains and any residual stresses, is an important consideration. The presence of non-metallic inclusions and second phase particles is also important in this respect.

8.1.3 On occasion, stress corrosion test programmes are mounted as the result of service failures and the failed components are used as a source of material. Unless it is desired to take specimens from cracked areas in order to examine the development of micro-cracks, it is important to ensure that specimens are only taken from established "crack-free" areas. Any variation in material structure within the component must be taken into account when selecting material for the manufacture of specimens.

8.1.4 It is desirable that identification marks or numbers should be permanently inscribed on test specimens. However, attention should be paid to their location on the specimen in order to avoid influencing the test result. They should be positioned as far away as possible from the test area, e.g. at the ends of bent-beam specimens.

8.1.5 Electric discharge machining (EDM) is commonly used in specimen preparation, for example compact tension specimens. This process generates hydrogen, which can be absorbed into the metal. Diffusible hydrogen would tend to degas with time but baking may be advisable though at a temperature that does not lead to any microstructural change. Hydrogen may still be fixed in irreversible deep traps and assessment of the possible impact should be undertaken though it is likely not to be important for most systems. The notch tip material will also be modified by EDM and a pre-crack should be grown beyond the depth of influence.

8.2 Surface condition

8.2.1 The initiation of stress corrosion cracks inevitably involves some initial surface reactions, and the surface condition of specimens may therefore exert a marked influence upon test results. The most obvious modification of surface finish is a variation in surface topography depending on the details of the preparatory technique, but it is also well established that residual stresses may be left in surfaces and that local changes in composition and structure may be associated with surface layers. It is important, therefore, in any testing programme to take these matters into account.

8.2.2 Surface topography would be expected to show a larger influence in harder notch-positive alloys or specimens of very small cross-section than in the softer more ductile materials or in large cross-section specimens, and this is found to be so. Thus, the stress corrosion cracking of brasses shows no significant variation with considerable change in surface topography, but grinding of high-strength steels, especially if carried out in such a manner as to induce micro-cracks, can produce a dramatic decrease in the cracking resistance. It is to be expected that the effect of variations in surfaces topography will vary inversely with cross-section of the specimen.

8.2.3 The question of which surface roughness parameter to use for stress corrosion cracking needs to be considered.

The surface roughness value (Ra) has been most commonly used but may not be the most relevant. It is defined in ISO 4287 as the “arithmetic mean of the absolute ordinate value $Z(x)$ within a sampling length”; i.e. an average surface roughness parameter reflecting the arithmetic mean displacement of the surface profile from the mean line. On cylindrical specimens that are ground longitudinally the value will be affected by the rate of longitudinal displacement of the grinding paper relative to the rotational speed of the specimen. Spiralling of score marks may occur if the relative ratio is not high and this will be reflected in the Ra value which otherwise may appear quite small. Because it is an average parameter, the Ra value may not identify specific surface features that could lead to stress corrosion cracking.

Stress corrosion initiation will be enhanced if the localized chemistry changes and the stress concentration associated with the surface profile can be maximised. In both cases, the sharpness of the profile is critical which is reflected to some extent in the width of the profile elements. For localized chemistry changes, the height of the profile (Z_t) is also critical and for the stress concentration, the depth of the valley (Z_v) is the key parameter. Stress corrosion cracking is likely with the optimum combination of these parameters allied to specific microchemical features of the material. Such a specific combination is not readily ascertained. Nevertheless, in addition to specifying Ra , there can be added value in evaluating and reporting the minimum and mean width of the profile elements, the maximum value of Z_t and the mean value (denoted Rc in ISO 4287), and the maximum and mean values of the valley depth.

ISO 4287 should be consulted for more exact definition of the various parameters.

8.2.4 Residual stresses may be left in surfaces as a result of inhomogeneous plastic deformation, e.g. arising from machining or indirectly because of thermal effects or volume changes associated with phase transformations. Local changes in composition may also occur. Residual stresses at the surface of stress corrosion test specimens are well known to influence lifetimes; compressive stresses increase and tensile stresses decrease the time to failure under otherwise comparable conditions. Residual stresses may be removed or minimised by appropriate heat treatment, provided that this is not likely to have other objectionable effects, e.g. upon mechanical properties.

8.2.5 Apart from any effects of residual stresses, the structural changes that may be induced in the surface layers of specimens may be significant in their own right in relation to the stress corrosion response of some alloys. Thus, the effects of plastic deformation by itself upon cracking resistance can be quite marked, and the localized phase transformations induced by deformation or the heat generated by the latter can also affect results. Such effects may account for the times to failure of 18 Cr/8 Ni steels being less (by a factor of about 4) with machined surfaces than with those produced by electropolishing, or for the increased susceptibility of specimens in high-strength, quenched and tempered steels, ground so as to promote the formation of a thin layer of untempered martensite at the surface.

8.2.6 Heat treatment of specimens after preparation is otherwise complete can produce barely perceptible changes in surface composition, e.g. decarburisation of steels or dezincification of brasses that promote quite dramatic changes in stress corrosion resistance. Similarly, oxide films, especially if formed at high temperatures during heat treatment or working, may be expected to influence stress corrosion test results, particularly when crack initiation constitutes a significant part of the lifetime.

8.2.7 When the final preparation of a test surface involves any chemical or electrochemical treatment, care shall be taken to minimize contamination by any residues from such treatments. In some cases, electropolishing is employed in order to overcome some of the difficulties associated with mechanical preparation, but the use of this technique can cause other problems. Chemical or electrochemical treatments that generate hydrogen must not be used on materials that are susceptible to hydrogen-induced damage. Such treatments can, in some cases, also result in selective phase attack which can influence test results.

8.2.8 Since a large proportion of the relatively few results that are available of the effects of surface finish have resulted from a few ad hoc experiments, rather than from systematically designed investigations, it

is difficult to do other than repeat that these effects should be borne in mind in interpreting results. To suggest that these effects can be avoided by the use of pre-cracked specimens is not always helpful for a variety of reasons, one of which is that in real engineering situations some of the effects mentioned are indeed present.

8.3 Area effects

Stress corrosion test results on some materials are dependent on the exposed area of the test specimen. Usually, the effect is on the scatter of results and it is therefore recommended that the specimen should be of sufficient size to minimize this effect.

8.4 Pre-cracked test pieces

8.4.1 Fracture mechanics is now commonly used in design and in evaluation of the significance of cracks detected in service. From a design viewpoint, this is recognition that crack-like defects may be introduced during fabrication or develop through service exposure. In the former case, the issue is whether these might develop as stress corrosion cracks; thus, the threshold stress intensity factor or mechanical driving force appropriate for short crack-like defects becomes important. Where crack propagation is anticipated or where cracks have been detected in service, the crack velocity is required to predict the expected or remaining life of the component of structure, or to guide inspection intervals.

NOTE When cracks are short, the plastic zone size may be of the same size as microstructural features such as grains and the adoption of continuum mechanics and linear elastic fracture mechanics becomes inappropriate.

8.4.2 The difficulties surrounding the choice of a plain specimen for assessing stress corrosion resistance may, at first sight, appear equally large in relation to pre-cracked specimens, in the sense that in the relatively short time during which such tests have been in use a large number of specimen types has been developed. However, the differing specimen geometries are related through the stress intensity factor, so that data from different tests can be compared, and the problem of choice is not, therefore, as difficult as may appear at first sight. The biggest single difficulty is in relation to the large size of specimen that is necessary for highly ductile materials if the concepts of linear elastic analysis are to be applied. Nevertheless, the use of pre-cracked specimens that dimensionally do not conform strictly to the requirements of linear elastic analysis is still worthwhile in some instances, provided that the results are only employed in service situations that involve a similar thickness.

8.4.3 It is important to recognize that pre-cracking is often by fatigue and inherently mostly transgranular. There can be long delays in initiating an intergranular crack in these circumstances and the threshold stress requirements may be different. Correspondingly, the threshold stress intensity factor determined by an increasing K experiment may be different from that from a decreasing K experiment as in the double cantilever beam test.

8.4.4 There has been a tendency in the past for some workers to regard tests on plain specimens as irrelevant and tests on pre-cracked specimens as the only ones likely to provide meaningful results; equally, there have been those with the diametrically opposed viewpoint although, fortunately, the numbers with such narrow viewpoints have diminished rapidly in recent years. It appears reasonable to suggest that in some cases when a stress corrosion crack has propagated some distance in a plain specimen the test becomes indistinguishable from one in which cracking is initiated from a pre-crack, at least in relation to the stress intensity, although there may be electrochemical differences between the two cases.

8.4.5 When estimating the length of the initial fatigue pre-crack prior to the commencement of stress corrosion testing, it should be appreciated that crack curvature may occur. Thus, the real maximum crack length may be greater than that estimated from measurements made on the specimen surfaces.

8.5 Welds

The distinctive features of welds are the differential material properties in the parent plate, heat affected zone and weld metal itself, and when testing in the as-welded condition the surface roughness, local stress concentrations and oxide film characteristics. Furthermore, there are likely to be residual stress and hardness variations through the thickness of the weld.

If the mechanical properties in the different zones vary significantly, differential straining may result under constant load. When testing in bend, e.g. 4-point bend, it is essential to strain gauge, usually in the parent plate as close to the weld as possible. Conventionally, the deflection is applied to give a desired strain in this region, corresponding to 100 % of the 0,2 % proof stress for example. The properties of the different zones may also change with temperature to different degrees and this poses unresolved problems of how best to stress the specimen. If the weld metal ductility decreases significantly with temperature, it may be difficult to achieve the desired strain at the strain gauge location unless the specimen is overloaded at ambient temperature. There is no ideal method in bend tests if the mechanical properties are markedly different at temperature.

Testing of fully machined weld specimens gives a defined surface finish but hardness and residual stress variations through-thickness mean that thought has to be given to the location of removal of the specimens. Repeat tests for welds are more critical than for homogenous plate.

In many respects, testing of fully machined specimens does not represent service performance and there is an increasing tendency to test in the as-welded state to account for surface roughness and stress concentrators for mechanical reasons. In addition, surface roughness and the as-welded oxide film can influence the corrosion response of the metal.

The adoption of fracture mechanics tests for welded specimens poses problems of defining the location of the notch. This is usually in the heat-affected zone but as the resistance of the material can vary from the fusion line to the parent plate, there is a need to take a statistical approach to this with notching at varying locations.

9 Stress corrosion test cells

9.1 The cell that contains the specimen and environment for stress corrosion tests frequently needs to be no more than a vessel made of some material, usually glass, that is inert to the environment and that produces no electrical response upon the test specimen. It is perhaps worth pointing out that, in addition to well-known effects such as the attack on glass vessels in concentrated NaOH solutions, less obvious interactions can occur. Such an example has been found in hot high-purity water environments where sufficient SiO₂ has been leached from standard laboratory glassware during tests to significantly affect the stress corrosion behaviour of low-alloy steel specimens.

9.2 Where cracking is initiated at surfaces through which heat transfer occurs, it may be necessary to design a cell in which such an effect is incorporated, since the concentration of substances in solution that may occur at an interface through which heat passes may play a significant role in promoting cracking, especially if surface deposits allow concentration by evaporation but prevent mixing with the bulk of the environment. Significant examples are the cracking of stainless steel piping under thermal insulation, the caustic cracking of riveted mild-steel boilers and cracking of stainless steel pipes or pressure vessels under dripping or spray conditions.

Methods have been developed for simulating conditions for cracking under these concentrating conditions, for example, the drop-evaporation test (ISO 15324).

9.3 The relationship, previously mentioned (see 6.3.5), between the area of test specimens and the volume of solution in which they are immersed, has obvious implications for the design of test cells.

10 Initiation of stress corrosion tests

It may be felt that the initiation of a stress corrosion test involves no more than bringing the environment into contact with the specimen in which a stress is generated, but the order in which these steps are carried out may influence the results, as may certain other actions at the start of the test. Thus, in outdoor exposure tests the time of the year at which the test is initiated can have a marked effect upon the time to failure; so, too, can the orientation of the specimen, i.e. according to whether the tension surface in bend specimens is horizontal upwards or downwards, or at some other angle. But even in laboratory tests, the time at which the stress is applied in relation to the time at which the specimen is exposed to the environment may influence results; the same applies to the time taken to reach test temperature or to apply any electrochemical stimulation. The observations, with respect to incremental loading of pre-cracked specimens, mentioned in [8.4.4](#) also have relevance in this context.

11 Assessment and treatment of results

11.1 As the number of stress corrosion test methods has increased over the years, so have the methods of assessment of results. From the original simple U-bend type of test, the results of which took the form of time to failure, more sophisticated techniques have emerged. [Annex A](#) summarizes the key results from the different test methods and typical engineering usage of these results.

11.2 In some cases, the individual effect of environment should be separately assessed using unstressed specimens to confirm whether the specimens under test have failed by stress corrosion cracking.

11.4 If it is required to minimize the number of specimens, a binary search procedure can be used to determine the threshold stress. The first test should be conducted at a particular initial stress, e.g. equal to half the yield strength of the material involved, with subsequent tests at other fractions of the yield strength according to a schedule such as that shown in the figure, depending upon whether or not failure occurred in preceding tests. For some systems, the tensile strength may be used as the reference framework. The principle is shown in [Figure 1](#).

11.5 In some test methods, time to the appearance of the first crack is the criterion used and this raises a number of points. Caution should be used when inspecting specimens during tests to avoid contaminating surfaces. Some solutions used for crack detection have been found to contain appreciable quantities of harmful impurities that may themselves promote stress corrosion cracking. It should be appreciated that replacing a specimen for re-exposure after removal for inspection can affect the final result and it is preferable to use several specimens at the same stress level to avoid the necessity for re-exposure.

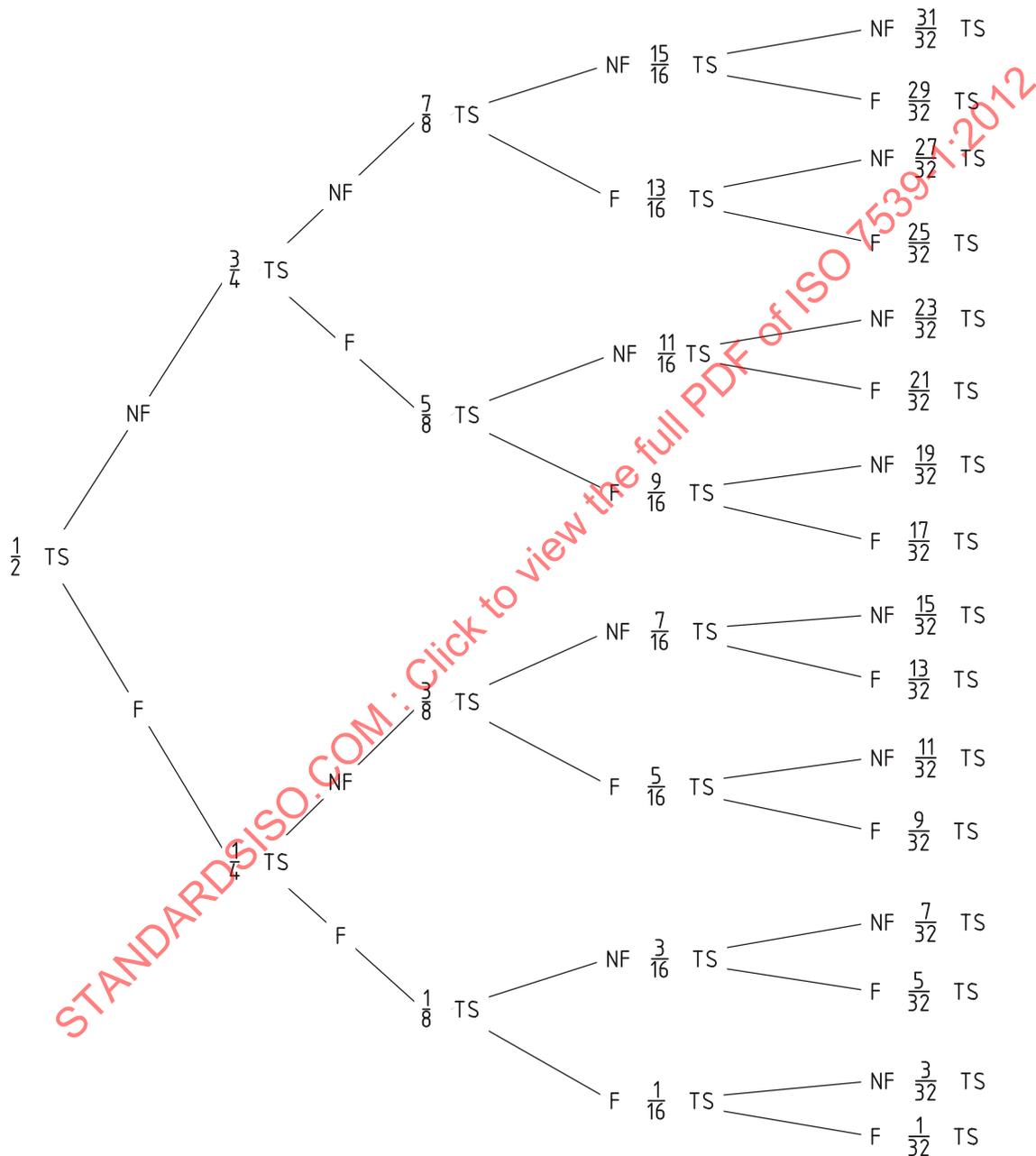
Often, low-power microscopy is used for examining specimens and in such cases a standard magnification (e.g. x 20) should be employed, since crack detection depends upon the resolving power of the system.

11.6 The results of slow strain rate tests can be assessed using a variety of parameters though most modern practice utilizes the plastic strain to failure. The elongation to fracture and reduction in area are commonly used. The maximum load achieved in the load-displacement curve is occasionally used though its sensitivity as a failure index can be limited unless significant work hardening is apparent. Occasionally, the appearance of the fracture surface has been used to assess susceptibility to stress corrosion, the percentage of environmental to ductile failure being used as the parameter.

11.7 Stress corrosion crack velocity or threshold stress intensity factor is often amongst the key information that is required by the design engineer and this can sometimes be determined within a relatively short period. The use of pre-cracked specimens is particularly suitable for estimation of crack velocity, with certain reservations. Crack extension can be monitored in a number of ways, e.g. compliance change, potential drop, etc.

NOTE Plain specimens have been used for determining crack velocity based upon the concept of interrupted testing in which crack depths after various time intervals can be obtained. However, in such tests, consideration has to be given to the need for the crack to traverse a significant microstructural distance. Also, such crack growth data are often crudely based on dividing the crack size by the exposure period and may not reflect the transient nature of the crack growth.

11.9 In common with most experimental investigations, stress corrosion test results are amenable to treatment using the normal statistical methods. Whenever possible, the results obtained should be validated in this manner.



Test 1	Test 2	Test 3	Test 4	Test 5