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**Test methods for stress corrosion
cracking on aluminium alloys**

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Reference number : JIS H 8711 : 2000 (E)

H 8711 : 2000 (ISO 9591 : 1992)

Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at the Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently **JIS H 8711 : 1990** is replaced with **JIS H 8711 : 2000**.

This revision puts stress on achieving conformance between the Japanese Industrial Standard and the International Standard, that is employed all the contents of the corresponding International Standard.

Attention is drawn to the possibility that some parts of this Standard may conflict with a patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have technical properties. The relevant Minister and the Japanese Industrial Standards Committee are not responsible for identifying the patent right, application for a patent after opening to the public, utility model right or application for registration of utility model after opening to the public which have the said technical properties.

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Test methods for stress corrosion cracking on aluminium alloys

Introduction This Japanese Industrial Standard has been prepared based on **ISO 9591 Corrosion of aluminium alloys—Determination of resistance to stress corrosion cracking** published in 1992, excluding annexes (informative) 1 to 7, without changing the technical contents.

For helping convenience for utility and understanding of this Standard, normative references to **ISO 9591**, **ISO 7539-1** to **ISO 7539-7** are employed as annexes (informative) 1 to 7 which are supplemented the text of this Standard.

In addition, "Informative references" with dotted underlines are the matters not stated in the original International Standards.

1 Scope

1.1 This Standard specifies a method for the determination of resistance to stress corrosion cracking (SCC) of aluminium alloys.

1.2 This Standard covers the method of sampling, the types of specimens, the loading procedure, the type of environment and the interpretation of results.

1.3 This Standard is aimed at the determination of the resistance to SCC as a function of the chemical composition, the method of manufacture and heat treatment of aluminium alloys.

1.4 This Standard applies to cast and wrought aluminium alloys in the form of castings, semi-finished products, parts and weldments.

1.5 Since most natural and many artificial environments contain chlorides, this Standard can be used to compare the performance of products employed under marine atmospheres and in environments containing chlorides providing that the failure mechanism is not changed. However, the result of this test should not be considered as an absolute criterion for the quality of alloys.

2 Normative references The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. If the indication of the year of coming into effect (or the year of publication) is given to these referred standards, only the edition of indicated year constitutes the provision of this Standard but the revision and amendment made thereafter are not applied. The normative references without the indication of the year of coming into effect (or the year of publication) apply limit only to the most recent edition (including the amendment).

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

ISO 7539-2 : 1989, *Corrosion of metals and alloys—Stress corrosion testing—Part 2 : Preparation and use of bent-beam specimens.*

ISO 7539-3 : 1989, *Corrosion of metals and alloys—Stress corrosion testing—Part 3 : Preparation and use of U-bend specimens.*

ISO 7539-4 : 1989, *Corrosion of metals and alloys—Stress corrosion testing—Part 4 : Preparation and use of uniaxially loaded tension specimens.*

ISO 7539-5 : 1989, *Corrosion of metals and alloys—Stress corrosion testing—Part 5 : Preparation and use of C-ring specimens.*

ISO 7539-6 : 1989, *Corrosion of metals and alloys—Stress corrosion testing—Part 6 : Preparation and use of pre-cracked specimens.*

ISO 7539-7 : 1989, *Corrosion of metals and alloys—Stress corrosion testing—Part 7 : Slow strain rate testing.*

Informative reference : Reference terms are specified in **JIS Z 0103** *Glossary of terms used in rust and corrosion preventive technology* and **JIS H 0201** *Glossary of terms used in the surface treatment of aluminium.*

3 Definitions For the purposes of this Standard, the definitions given in **ISO 7539-1** (refer to annex 1) apply.

Informative reference : Reference terms are specified in **JIS Z 0103** and **JIS H 0201**.

4 General principles

4.1 This Standard specifies two methods of loading:

- under constant total strain;
- under constant load.

4.2 This Standard specifies two methods of immersion in the solution:

- a) alternate immersion;
- b) continuous immersion (subject to agreement between the interested parties).

4.3 The evaluation criteria for corrosion cracking of alloys are

- a) σ_{SCC} : the threshold stress, which is the maximum stress under which no failure of the samples occurs during the fixed period of the test;
- b) τ_{SCC} : the time of failure, which is the moment of the appearance of the first visible crack (or under magnification up to $\times 30$) for specimens under constant strain.

4.4 The selection of the method of loading, the value of stresses, corrosive environment and criteria of evaluation can be the subject of an agreement between the interested parties and should be defined by the test programme.

5 Apparatus and materials

5.1 Loading apparatus Tensile stresses in the specimens are produced with yokes, stressing screws, springs, lever devices and special testing machines.

5.2 Construction materials If in contact with the salt solution, they shall not be affected by the corrodent to such an extent that they can cause contamination of the solution and change its corrosiveness.

5.2.1 Use of inert plastics or glass is recommended where feasible.

5.2.2 Metallic components which are in contact with the solution should be made from corrosion-resistant alloys (e.g. titanium or stainless steel), or protected with a suitable corrosion-resistant coating that also satisfies 5.2.

5.3 Specimen holders They should be designed to electrically insulate the specimens from each other and from any bare metal parts. When this is not possible, as in the case of certain stressing bolts or jigs, the bare metal contacting the specimen should be isolated from the corrodent by a suitable coating. Protective coatings should be of a type that will not leach inhibiting or accelerating ions or protective oils or leave any residue, e.g. vapour, on the non-coated portions of the specimen. In particular, coatings containing chromates should be avoided. It is recommended that all samples should be degreased after coating.

5.4 Apparatus for alternate immersion in solutions

5.4.1 Any suitable mechanism may be used to accomplish the immersion portion of the cycle provided that:

- a) it achieves the specified rate of immersion and removal; and
- b) the apparatus is constructed of suitable inert materials.

The usual methods of alternate immersion are

- a) specimens are placed on a movable rack that is periodically lowered into a stationary tank containing the solution;
- b) specimens are placed on a corrosion wheel arrangement which rotates every 10 min through 60° and thereby passes the specimens through a stationary tank of solution;
- c) specimens are placed in a stationary tray open to the atmosphere and the solution is moved by air pressure, by a non-metallic pump, or by gravity drain from the reservoir to the tray.

5.4.2 The rate of immersion and removal of the specimens from the solution should be as rapid as possible without jarring them. For purposes of standardization, an arbitrary limit shall be adopted such that no more than 2 min elapse from the time of immersion in the solution or exhibiting in the air.

6 Sampling

6.1 In general, this International Standard specifies three specimen orientations for thick products and two for thin products. The orientation diagram is given in figure 1 in annex A. In figure 1a) in annex A, the first direction refers to the specimen axis and the second direction refers to the direction of crack growth.

6.2 Unless otherwise specified, tests should be performed in the short transverse direction (S) for thick products and in the long transverse direction (T) for thin products. In rolled or extruded sections that are approximately round or square, the samples should be oriented in the transverse (diametrical) direction.

In the case of forgings and, more generally, when the structure of the test pieces is not obvious, it is recommended that the grain direction be determined by macroetching or by metallographic examination, in order to select specimens in the most susceptible (short transverse or long transverse) directions (see annex A).

6.3 The number of specimens to be submitted to testing should be agreed between the interested parties, but for each case at one stress level it is recommended that no less than four adjacent specimens shall be tested, and at several testing levels, no less than three specimens.

7 Specimens

7.1 Type and sizes Specimens as defined by ISO 7539-2 to ISO 7539-5 can be used (refer to annexes 2 to 5).

7.1.1 Tension specimens, C-ring specimens or bend specimens can be taken from thick products, for example from plates or forgings.

7.1.2 Tension specimens, bent beam specimens or U-bend specimens can be taken from thin produces, for example from sheets.

7.1.3 When specimens are taken from weldments where the weld is perpendicular to the stress axis, the weld should be placed in the middle of the specimen.

7.1.4 The comparison of different alloys and tempers should be conducted on specimens of the same by and size. Where possible, specimens should be heat treated before final machining, otherwise consideration must be given to the removal of oxidation products from the surface (see 7.2.4).

7.2 Surface preparation

7.2.1 Specimens with surface defects (of mechanical or metallurgical origin) should be discarded.

7.2.2 The surface quality of a specimen should comply with the following.

- a) without mechanical machining, in the as-supplied condition;
- b) with mechanical machining: the arithmetical mean deviation of the profile (R_a) less than or equal to 1 μm is recommended, unless it is required to simulate an as-manufactured surface condition;
- c) the surface condition of welded specimens should be agreed between the interested parties.

7.2.3 Machining practices should not alter the metallurgical structure of the specimens and should be designed to minimize the generation of residual stresses.

7.2.4 Test specimens should be degreased with an organic solvent (e.g. petroleum ether or acetone). Alternative surface treatments, for example pickling or etching, may be agreed between the interested parties. However, it should be recognized that chemical treatments may induce crack initiation in some tempers and alloys. In this case chemical treatment is not recommended.

7.3 Specimen identification Specimens should be identified by a suitable method (see ISO 7539-1, refer to annex 1) and the markings should be protected against corrosion.

7.4 Precautions After degreasing or pickling, the test region of the specimens should not be touched prior to testing.

8 Test environments The test environment should be relevant to the intended application. Unless otherwise agreed, the following environments are recommended.

8.1 Analytical grade chemicals are recommended for the preparation of the corrosive solutions.

Informative reference : Analytical grade chemicals refer to reagents specified in JISs.

8.2 The solution should be prepared using distilled or deionized water. The minimum resistivity should be $10^5 \Omega \cdot \text{cm}$.

8.3 For alternate immersions, an aqueous $3.5 \% (m/m) \pm 1 \% (m/m)$ sodium chloride solution should be used. The pH of this solution, when freshly prepared for the test, shall be between 6.4 and 7.2, following adjustment, if necessary (see 8.5).

8.4 For continuous immersion, an aqueous solution $2.0 \% (m/m) \pm 0.1 \% (m/m)$ sodium chloride and $0.5 \% (m/m) \pm 0.05 \% (m/m)$ sodium chromate (Na_2CrO_4) should be used.

The pH of this solution should be adjusted to within 3.0 ± 0.2 with hydrochloric acid. The pH should be checked regularly during the test and adjusted, if necessary (see 8.5).

8.5 Only diluted hydrochloric acid or sodium hydroxide solution should be used to adjust the pH of test solutions.

8.6 The temperature and humidity of the laboratory should be controlled to ensure that the samples dry out between consecutive immersion periods during alternate immersion testing.

The relative humidity of the air should be controlled and maintained between 40 % and 75 %.

A temperature of $25^\circ\text{C} \pm 3^\circ\text{C}$ is recommended to ensure the reproducibility of results.

8.7 Before the freshly prepared solution is used, its temperature should be within 3°C of the specified air temperature. Thereafter, no control of the solution temperature is required. Instead, the ambient temperature is controlled and the solution is allowed to reach temperature equilibrium.

8.8 The minimum ratio between the volume of test solution and the exposed area of specimens (including any uncoated accessories made from aluminium alloys) should be 35 ml/cm² for alloys exposed under alternate immersion conditions, and 10 ml/cm² for alloys exposed under continuous immersion conditions in sodium chloride/sodium chromate solution.

8.9 Evaporation losses should be made up by daily additions of water of the required purity (see 8.2).

8.10 Fresh solution should be prepared weekly. At the same time, the portions of the apparatus in contact with the solution should be cleaned by flushing with water.

9 Stress considerations The loads should be such that the stresses are elastic. The elastic stresses are calculated from the equations given in ISO 7539-2 to ISO 7539-5 (refer to annexes 2 to 5)

10 Procedure

10.1 The specimens should be exposed to the test environment as soon as possible after stressing.

10.2 During alternate immersion, the specimens are totally immersed in the sodium chloride solution for 10 min of each hour and then removed from the solution and allowed to dry for 50 min.

A mild circulation of air is recommended and stagnant air conditions should be avoided.

Drying by forced air blast on the specimens is not recommended because of the difficulty in maintaining uniform drying of large groups of specimens.

The tests are usually carried out without interruption during a given exposure time.

10.3 The test duration will depend upon the composition and heat treatment of the alloy, the specimen size, the environment and the method of stressing. Usually, test durations of between 10 days and 90 days are employed.

When the purpose of the test is to control the quality of production lots or to determine the characteristics laid down in the standard documents concerning alloys, the test duration shall be as required in pertinent specifications or shall form the subject of an agreement between the interested parties.

10.4 It is forbidden to test simultaneously specimens of dissimilar alloy composition in the same salt solution.

For maximum reproducibility, do not expose specimens of low alloy content to the solution used for specimens of high alloy content if the alloying element differs appreciably from the base metal in the galvanic series in salt water. For example, specimens of copper-free aluminium alloys should not be exposed with specimens from alloys containing more than about 0.5 % copper.

10.5 After exposure, unfailed specimens under constant strain should be unloaded, rinsed with water, and then cleaned in concentrated nitric acid (60 % to 70 %) at room temperature to remove corrosion products. They should then be rinsed with water and dried prior to more detailed examination.

11. Assessment of results

11.1 Test specimens exposed under constant strain, which show questionable corrosion after cleaning, may require metallographic examination. For specimens under constant load, a metallographic examination may be used for failures occurring after more than 10 days, to check if failure is due to stress corrosion or to another form of corrosion. A specimen that reveals only transgranular pitting corrosion or pitting plus ductile failure shall not be considered as an SCC failure.

11.2 Optical metallographic examination of a cross-section may be used to distinguish between stress corrosion, intergranular corrosion and transgranular pits. A specimen that reveals intergranular cracks deeper than transgranular pits shall be considered as an SCC failure. Intergranular corrosion not deeper than transgranular pits shall not be considered as an SCC failure.

For specimens under constant load; scanning electron microscope (SEM) examination of the fracture surface allows brittle intergranular failure due to stress corrosion to be distinguished from transgranular ductile failures due to mechanical overload. Both type of failures are present when SCC is the origin of failures for specimens under constant load.

12 Test report The test report should contain the following information:

- a) the chemical composition or designation of the alloy;
- b) product, temper and section thickness of material tested;
- c) size and location of specimens;
- d) type, size and grain orientation of test specimen and number of replicates;
- e) level(s) of stress and method of loading;
- f) surface condition of the specimens;
- g) duration of test or time to failure of individual specimens;
- h) test method and condition of environment;
- i) results of any metallographic examinations.

Annex A (normative)

Grain orientation examination

A.1 Macroetching Conduct etching on a machined surface.

Immerse for about 1 min at room temperature in a solution composed of

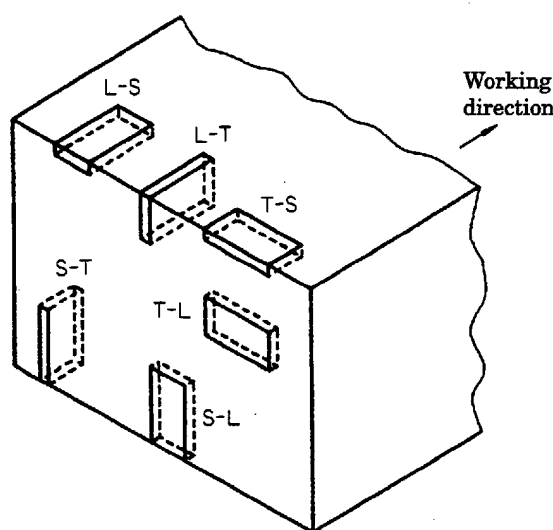
- a) 1 volume hydrochloric acid, 38 % (m/m), $\rho = 1.19$ g/ml;
- b) 1 volume of nitric acid, 60 % (m/m), $\rho = 1.38$ g/ml;
- c) 2 % (V/V) of hydrofluoric acid, 40 % (m/m), $\rho = 1.14$ g/ml; and make up to 3 volumes with water.

Visually inspect with the naked eye or at low magnifications.

A.2 Fluoboric etching Conduct etching on a carefully polished surface, preferably after electrolytic polishing.

- a) Anodic oxidation: 20 mA/cm² in an aqueous solution of 2.5 % (V/V) fluoboric acid for 1 min or 2 min at room temperature.

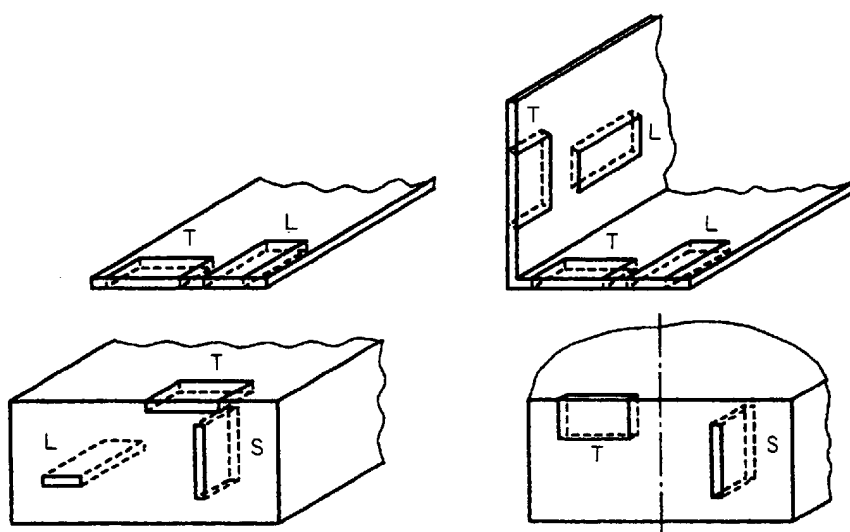
Examine using a microscope in polarized light.



- L—T Longitudinal—Long transverse direction
- L—S Longitudinal—Short transverse direction
- T—L Long transverse—Longitudinal direction
- T—S Long transverse—Short transverse direction
- S—L Short transverse—Longitudinal direction
- S—T Short transverse—Long transverse direction

a) General procedure

Figure 1 — Specimen orientation



L Longitudinal direction
 T Long transverse direction
 S Short transverse direction

b) Recommended procedure

Figure 1 (concluded)

Annex 1 (informative)**Corrosion of metals and alloys—Stress corrosion testing—
Part 1 : General guidance on testing procedures**

This annex (informative) supplements the text and annex A (normative), and is not part of the specifications. This annex has been prepared based on **ISO 7539-1 : 1987** which is quoted in this Standard, for the sake of convenience for users of this Standard.

0 Introduction This part of **ISO 7539** gives general guidance on the selection, use and interpretation of the significance of various test procedures that have been developed for the assessment of the resistance of metals and alloys to stress corrosion. These test procedures are described in a series of additional parts as follows:

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*

Part 7 : *Slow strain rate testing*

1 Scope This part of **ISO 7539** describes the general considerations which apply when designing and conducting tests to assess susceptibility of metals to stress corrosion.

NOTE — Particular methods of test are not treated in detail in this document. These are described in the additional parts of **ISO 7539**.

2 Definitions

2.1 stress corrosion : Synergistic attack on a metal caused by the simultaneous action of a corrosive environment and nominally static tensile stress which usually results in the formation of cracks. This process frequently results in a significant reduction of the load-bearing properties of metallic structures.

NOTE — See stress corrosion cracking (3.1).

2.2 threshold stress (for stress corrosion) : The stress above which stress corrosion cracks initiate and grow, for the specified test conditions.

2.3 threshold stress intensity factor (for stress corrosion) : The stress intensity factor above which stress corrosion cracks will initiate, under conditions of high constraint to plastic deformation, i.e. under plane strain predominant conditions.

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. In the case of stress corrosion the environment is often quite specific (see 6).

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

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

2.7 time to failure : The 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 : A test involving controlled extension or bending of the test specimen at a strain rate usually in the region 10^{-3} to 10^{-7} s⁻¹. The strain is increased either continuously or in steps, but not cyclically.

2.9 average crack velocity : The maximum depth of crack(s) due to stress corrosion, divided by the test time.

2.10 orientation : The 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 From the definition of stress corrosion (2.1) it is apparent that stress corrosion cracking is a particular case of stress corrosion and in some circumstances attack may not result in the formation of cracks. Although it is generally agreed that cracking is the usual result, other manifestations such as intergranular corrosion or elongated fissures, which are enhanced by the presence of stress, have also to be recognized.

Whilst recognizing that these differences exist, for the purpose of this document, which is concerned with test methods, the terms "stress corrosion" and "stress corrosion cracking" can be regarded as being synonymous as is usually the case in corrosion literature.

As far as this International Standard 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.

NOTE — A distinction should be made between local dissolution due to deteriorations and those phenomena caused by hydrogen. The two types of phenomena may be superimposed but cannot be confused with a phenomenon directly imputable to deliberate hydrogen loading.

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 It is important to realize that the word "test" has a special meaning in the context of stress corrosion resistance or susceptibility. Whether or not a stress corrosion process occurs in a given case depends on both the exposure conditions and the properties of the material. The word "susceptibility" to stress corrosion does not describe a material property or quality that can be located on a universally applicable scale, since the order of merit of a given set of alloys may vary with exposure conditions.

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 International Standard 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 this International Standard 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. In preparing this part, use has been made of an earlier review of the subject, updated where appropriate.

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. Whilst 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.

5 Stressing systems

5.1 General Methods of loading test pieces, whether initially plain, 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 strain 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 problems with such test methods are usually related to poor reproducibility of the stress level, if indeed any quantitative measure of this is available. Attempts to improve upon this situation have led to more sophisticated types of bend test, e.g. involving four-instead of three-point loading, but the limitations of simple bending theory, usually used to calculate the stress level, can lead to errors in anticipated stress especially when strains beyond the elastic limit are required. The use of strain gauges for measuring surface stresses may be useful in some circumstances. The fabrication of strip specimens to produce "U" bends introduces significant amounts of plastic deformation which may influence cracking response.

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, thus simplifying both the application and calculation of the stress. However, the former require more massive restraining frames than bend test specimens of similar cross-section.

5.2.6 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, but 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 and where welding is involved the structural modifications may raise difficulties, unless the test is simulative of a practical situation.

5.2.7 Constant total strain specimens are sometimes loaded by being placed initially into conventional testing machines or similar devices and then, whilst being maintained in their 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 the specimen remains constant as the restraint is transferred from the testing machine to the frame. This implies a similar stiffness in the testing machine and frame, which is likely to be so only if the frame is relatively massive compared with the specimens.

5.2.8 The stiffness of the stressing frame employed, may also influence the time to failure of a specimen, quite apart from any effect that it may have upon the initial stress level. Thus, in most constant total strain tests and especially those upon ductile materials, the initial elastic strain in the specimen is converted in part to plastic strain as the crack propagates.

5.2.9 Once load relaxation has been initiated, the extent to which it proceeds can vary from specimen to specimen and this may influence time to failure according to the number of cracks or pits that develop. Marked load relaxation can be observed on a specimen with many cracks or pits whereas little load relaxation is observed when only a few cracks are present. If only one crack develops, it will not need to grow to large dimensions before sudden, final failure occurs because the applied load remains high, whereas the marked load relaxation associated with the presence of many stress corrosion cracks means that they must propagate much further before one of them becomes large enough to create the stress conditions, at a relatively small load, for sudden failure.

5.2.10 The extent to which the number of cracks present influences the test results naturally depends upon the stress corrosion system being studied, i.e. upon such properties as the fracture toughness of the material and even upon the aggressiveness of

the environment employed. The result also depends upon the stiffness of the restraining jig employed. Thus the stiffer the frame the less the elastic strain that is likely to remain in the specimen after the propagation of a Luders band. Therefore the time to failure for a given initial stress varies according to whether the system is hard or soft; in some cases cracks may cease propagating so that failure is not achieved.

5.3 Constant load tests

5.3.1 These may simulate more closely stress corrosion failure from applied or working stresses. Since the effective cross-section of the test piece is reduced by crack propagation, constant load tests involve an increasing stress situation. Consequently, such tests are more likely to lead to early failure or total failure than are constant total strain tests.

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, say, 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 very small section specimens (see 7.2.2).

5.3.4 The cost of testing specimens under constant load on individual testing machines can be minimized 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 which would otherwise be inevitable in the event of a failure. Users must invalidate test procedures employing chains of specimens to ensure freedom from errors before proceeding with their adoption.

5.3.5 The use of a tension specimen having a tapered gauge length, which is finding increasing popularity, 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 results may be influenced by such factors as number of cracks present, net section yielding, etc. It may be more appropriate to use such specimens in "sorting" 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 at stresses below the threshold stress. Thus, the threshold stress value is likely to be lower in a particular system when determined under constant load conditions than under constant deflection conditions.

5.4 Slow strain rate tests

5.4.1 The application of slow dynamic straining, which was initially regarded as a rapid sorting test, is beginning to emerge as one that has much more relevance to practice.

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 Stress corrosion crack velocities usually fall in the range of 10^{-3} to 10^{-6} mm/s , which implies that failures in laboratory tests under constant total strain or constant load conditions for specimens of usual dimensions occur in a few days. This is found to be so if the system is one in which stress corrosion cracks are readily initiated, but it is also common experience to find that test pieces do not fail in very extended periods of testing, which are then terminated at some arbitrary selected time. The consequences are that considerable scatter may be associated with replicate tests and the arbitrary termination of the test leaves an element of doubt concerning what the outcome would have been if it had been allowed to continue or a longer time. Just as the use of pre-cracked specimens assists in stress corrosion crack initiation, so apparently may the application of slow dynamic strain, which has the further advantage that the test is not terminated after some arbitrary time, since the conclusion is always achieved by the specimen fracturing and the criterion of cracking is then related to the mode of failure. Thus, in the form in which it is normally employed, the slow strain rate method frequently results in failure within about 2 days, either by ductile fracture or by stress corrosion cracking, according to the susceptibility towards the latter. The fact that the test concludes in this positive manner in a relatively short period of time constitutes one of its main attractions.

5.4.3 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 and in this respect it is in a similar category to tests on pre-cracked specimens. In recent years an understanding of the implications of dynamic strain testing has developed and it now appears that this type of test may have more relevance and significance than just that of an effective, rapid, sorting test. It may be argued that laboratory tests involving the pulling of specimens to failure at a slow strain rate show little relation to the reality of service failures. In fact, in both constant total strain and constant load tests crack propagation also occurs under conditions of slow dynamic strain to a greater or lesser degree depending upon the initial value of stress, the time at which a crack is initiated and various metallurgical

parameters that govern creep in the specimen. Moreover, there is an increasing amount of evidence in some systems which suggests that the function of stress in stress corrosion is to promote a strain rate which, rather than the stress as such, is the important mechanical parameter in crack initiation or propagation. In these cases the minimum creep rate for cracking is as much an engineering design parameter as is the threshold stress or stress intensity factor obtained from constant load tests on plain or pre-cracked specimens.

5.4.4 The equipment required for slow strain testing is simply a device which permits a selection of strain rates whilst 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^{-7} mm/s.

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.

5.4.5 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.

6 Environmental aspects

6.1 General Stress corrosion cracking is still considered to occur in rather specific alloy/environment combinations, e.g. austenitic stainless steels in chloride solutions and mild steels in nitrate solutions. However, the list of such combinations continues to grow with time and even instances of the cracking of materials in high purity water have been observed. Moreover, it should be noted that substances in the gaseous phase may also influence stress corrosion mechanisms and gaseous environments are sometimes used for testing. In such cases pressure is likely to be an important parameter.

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. Whilst 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 common rate due, for example, to the more rapid formation of protective films. Similarly, the reduction in the solubility of oxygen in aqueous solutions, which may accompany an increase in temperature, can also result in a lower corrosion rate, and other examples could be quoted.

Such effects are likely to be especially relevant in the case of stress corrosion which, as previously mentioned, occurs under rather specific conditions often involving a balance between active corrosion and passive behaviour.

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. 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 Solution composition

6.3.1 Although it is inevitable that the environment remains as one of the very important variables in stress corrosion testing, some solutions have become particularly widely used for certain types of alloy. Boiling magnesium chloride solutions for stainless steels and boiling nitrate solutions for carbon steels are two examples. Such solutions have been criticized for several reasons, the main one being that they do not generally reproduce plant conditions. This may be of considerable importance in that the relative cracking susceptibilities of a range of alloys are not necessarily the same in different environments.

6.3.2 Nevertheless, tests in these commonly used solutions can serve a useful purpose, provided that their limitations are borne in mind and that adequate care is taken in preparing and using the solutions. Whilst the relatively small differences that may be expected to occur between laboratories preparing a solution to the same specification would frequently not influence stress corrosion test results, 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.

6.3.3 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 electrodes 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.

6.3.4 Where oxygen plays an important part in the corrosion reactions that promote cracking, small changes in oxygen concentration can play an influential role. Thus, in the testing of certain aluminium alloys in aerated solutions, failure may occur in several hours, but in deaerated solutions no cracking may result in extended tests. Oxygen put into or taken from a solution as an intentional exercise is indicative of concern for possible effects arising from its presence. The entrainment of oxygen in a solution by stirring or spraying is reflected in much shorter failure times in aluminium alloys subjected to spraying than when totally immersed.

6.3.5 As was mentioned earlier, the results from tests in one of the commonly used solutions are sometimes assumed to indicate the relative susceptibilities of a range of alloys irrespective of the environments to which these may be ultimately subjected in service. The danger in drawing conclusions from tests in a given environment and applying these to a different situation indicates the need to simulate service conditions as closely as possible if laboratory data are to be used for selection or design in relation to industrial equipment.

6.3.6 In relation to simulating service environment 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. Another example is provided by pitting that develops as a precursor to stress corrosion cracking, in that the cracking environment proper often develops during the pitting stage. Similarly, it should be appreciated that the environment at the tip of a crack may differ from the bulk environment. This applies to both pre-cracked specimens and the tips of cracks growing in initially plain specimens.

6.4 Electrochemical aspects

6.4.1 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. It has sometimes been assumed that movement of the potential in the anodic direction will increase susceptibility to cracking whilst the application of cathodic current will retard cracking or prevent it altogether. Depending upon the details of the cracking mechanism, this may or may not be true. For example, if the alloy is susceptible to cracking resulting from the influx of hydrogen, the effects of changing the potential may be opposite to that mentioned above, in which it is assumed that the cracking involves dissolution along an active path. The usual reason for increasing current or controlling the potential of stress corrosion test specimens, where data collection is the objective, is to reduce the time to failure in laboratory tests or to improve reproducibility. It should not be assumed that if a galvanostatic technique is used the effect will simply be to influence the kinetics of cracking, since the applied current is also likely to alter the potential and this could promote a different response. It is recommended therefore that electrochemical stimulation of stress corrosion tests should not be used unless adequate care is taken to ensure that the mechanism of failure is not changed from that which operates at the free corrosion potential and that the data correlate well with service experience.

6.4.2 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.

6.4.3 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.

6.4.4 When it is established that stress corrosion occurs only over a critical range of potentials it is possible to monitor by on-line potential measurements whether cracking is 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.

6.4.5 The use of a potentiostat in the laboratory tests, whilst 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. Galvanostatic techniques, which can involve lower costs than those employing a potentiostat, may also be useful in some situations but the current density applied should be relatively small so as not to move the potential far from the free corrosion potential, unless it is known that any of the effects of potential mentioned above are not relevant to the system being studied. 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, say, 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 is not considered adequate, is to conduct a properly designed and statistically significant set of experiments.

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

6.4.7 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 Specimen design and manufacture

7.1 General

7.1.1 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 indeed 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.

7.1.2 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.

7.1.3 On occasions, 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 selection material for the manufacture of specimens.

7.1.4 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.

7.2 Surface condition

7.2.1 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.

7.2.2 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.

7.2.3 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 minimized by appropriate heat treatment, provided that this is not likely to have other objectionable effects, e.g. upon mechanical properties.

7.2.4 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 increase 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.

7.2.5 Heat treatment of specimens after preparation is otherwise complete can produce barely perceptible changes in surface composition, e.g. decarburization 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.

7.2.6 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.

7.2.7 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, of course, that in real engineering situations some of the effects mentioned are indeed present.

7.3 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.

7.4 Pre-cracked test pieces

7.4.1 The literature contains many references to the use of notched, as opposed to plain, specimens in laboratory studies of stress corrosion, for reasons of improved reproducibility, inability to crack plain specimens under otherwise identical conditions, or ease of measuring some parameter such as crack growth rate if the crack location is predetermined. However, developments in fracture mechanics have resulted in the evolution of a whole new field of stress corrosion testing involving the use of specimens containing a sharp pre-crack, usually produced from a notch by fatigue loading. For present purposes it is sufficient to point out that this approach involves a parameter K , the stress intensity factor, that defines the stress field at the leading edge of a crack. This concept is of considerable practical importance, especially in relation to higher yield strength materials, since engineering structures frequently contain flaws, produced during fabrication or service, the significance of which may be assessed in relation to whether they are likely to propagate as stress corrosion cracks, or, if they do, to what extent they may be allowed to propagate before the structure is deemed unsafe.

7.4.2 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. Since it is probable that most service stress corrosion failures occur in highly ductile materials in relatively thin sections, it is clear that there are problems here; 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 similar thicknesses.

7.4.3 Quite apart from fracture mechanics considerations there are sometimes instances where the use of pre-cracked specimens may be justified in relation to the simulation of service situations, to the relative ease with which stress corrosion cracks can be initiated at pre-cracks or to the advantages that sometimes accrue from the propagation of a single crack. The claim that has sometimes been made for pre-cracked specimen tests — that they circumvent the initiation stage of cracking in plain specimens, erroneously assumed to be invariably related to the creation of a corrosion pit that provides a measure of stress concentration approaching that achieved at the outset with a pre-crack — is rarely entirely valid. Thus, the geometries of a pit, notch or pre-crack are frequently as important for electrochemical reasons as they are for any reason associated with their influences upon stress distribution. This is because a geometrical discontinuity may be necessary to provide the localized electrochemical conditions, in terms of environment composition or electrode potential, that are necessary for stress corrosion crack propagation. The objections that have sometimes been made against the use of pre-cracked specimens, e.g. on the validity of introducing a transgranular pre-crack into a specimen that suffers intergranular stress corrosion cracking, or on the necessity to go to considerable expense

to produce a very sharp crack when the introduction of a corrosive environment may blunt the crack by dissolution, miss the point that such sharp discontinuities do indeed exist in real materials. Indeed, one of the major attractions of pre-cracked specimen testing is that it can provide data from which can be calculated the maximum allowable defect sizes in structures for the latter to remain in a safe condition. It should, however, be appreciated that there exist lower limits of defects size and upper limits of applied stress beyond which the concepts of fracture mechanics are no longer relevant. In particular, the application of fracture mechanics to defect sizes below about 0.1 mm, or when the local stresses are approaching the yield stress, must be treated with caution since under these circumstances the predictions of maximum allowable defect size may no longer be reliable.

7.4.4 One aspect of the testing technique with the use of pre-cracked specimens, where there appear to be some differences in the experience of different laboratories, is concerned with the use of incremental loading to determine the threshold intensity factor. Whilst close correlation between the values obtained by step loading and direct loading has been reported, some dependence upon loading rate in the former method has also been quoted.

7.4.5 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 view-point although, fortunately, the numbers with such narrow view-points 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.

7.4.6 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 Stress corrosion test cells

8.1 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.

8.2 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 and the caustic cracking of riveted mild-steel boilers.

Methods have been developed for simulating conditions for cracking under these concentrating conditions.

8.3 The relationship, previously mentioned (see **6.3.3**), 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.

9 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 **7.4.4** also have relevance in this context.

10 Assessment and treatment of results

10.1 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.

Just as the selection of a test method depends upon a variety of factors, so does the method of assessment and it is acknowledged that in some cases only a fairly simple appraisal is possible. Nevertheless, it is important that the limitations of the particular techniques are appreciated.

10.2 The earliest method of test evaluation was the time to failure (or no failure) of the specimen and this is still quite widely used. The limitations of such an approach, e.g. effects of stressing frame stiffness, fracture toughness of material, aggressiveness of environment, number of cracks initiated, specimen thickness, etc., mentioned earlier (see **5.2**) should be borne in mind.

10.3 It is common practice to compare materials, from a stress corrosion resistance view-point, in terms of their times to failure for a given stress, but even using identical equipment this may not always be justifiable. A more satisfactory basis for comparison would seem to be the threshold stress level or, in the case of pre-cracked specimens, the threshold stress intensity level. This requires the determination of the appropriate stress ν , failure time curve. It is preferable to expose specimens over a range of initial stress/stress intensity levels rather than to conduct a large number of replicate tests at the same stress/stress intensity level. However, even using this approach some variation in results might be expected depending upon the type of test. The threshold stress level under constant load testing, for example, tends frequently to be lower than obtained under constant total strain conditions. In addition, the results depend upon the test environment as the threshold obtained in one solution cannot be assumed to be applicable to other cases.

10.4 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 tensile strength of the material involved, with subsequent tests at other fractions of the tensile strength according to a schedule such as that shown in the figure, depending upon whether or not failure occurred in preceding tests.

10.5 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 (say $\times 20$) should be employed, since crack detection depends upon the resolving power of the system.

10.6 From the definition of stress corrosion it is clear that conjoint action is involved, and therefore the individual effect of environment should be separately assessed to confirm whether the specimens under test have failed by stress corrosion cracking. Test programmes should include the exposure of unstressed specimens which can be examined by various means at the determination of the test for comparison purposes.

10.7 The results of slow strain rate tests can be assessed using a variety of parameters. The effects of stress corrosion cracking may be reflected in the load-deflection curve either in terms of maximum load achieved or elongation to fracture. These may be used, therefore, for expressing cracking susceptibility, as may also the reduction in area. In some instances a combination of load and ductility may provide a useful basis for comparison. As with other test methods, time to failure is often a useful method of assessment, the results usually being normalized by dividing them by the time to failure at the same strain rate in a test carried out in an inert environment at the same temperature. 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.

10.8 Stress corrosion crack velocity or threshold intensity factor is sometimes the important information which is required by the design engineer and this can be determined in several days. 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, acoustic emission, potential drop, X-rays, etc. Plain specimens can also be used for determining crack velocity based upon the concept of interrupted testing in which crack depths after various time intervals can be obtained. Threshold stress intensity factors can also be derived from the above observations or indeed by incremental loading of pre-cracked specimens.

10.9 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.

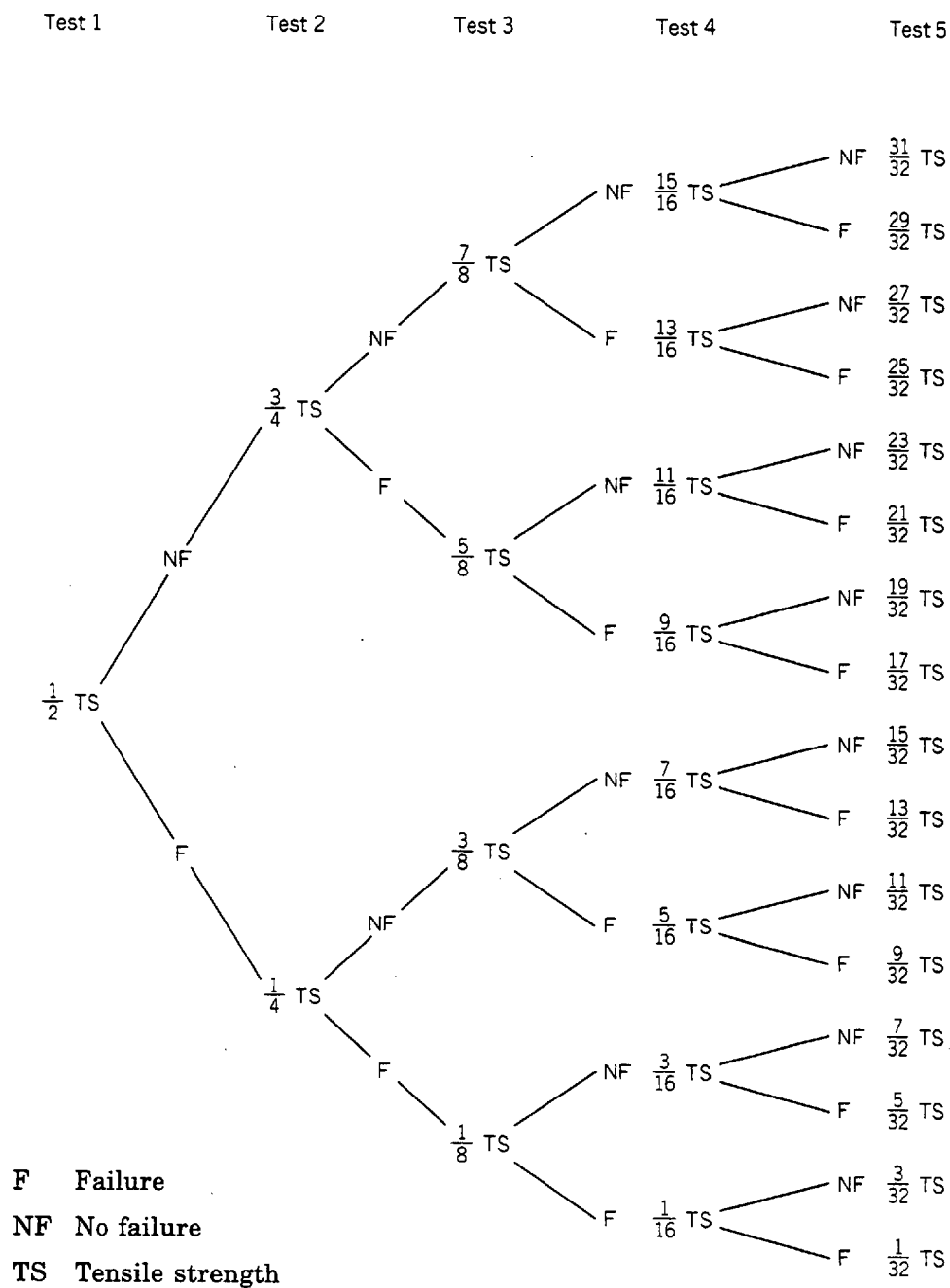


Figure — Binary search procedure to determine threshold stress

Annex 2 (informative)**Corrosion of metals and alloys—Stress corrosion testing—
Part 2 : Preparation and use of bent-beam specimens**

This annex (informative) supplements the text and annex A (normative), and is not part of the specifications. This annex has been prepared based on **ISO 7539-2 : 1989** which is quoted in this Standard, for the sake of convenience for users of this Standard.

WARNING — Bent-beam specimens made from high strength materials may fracture rapidly; pieces may fly off at high velocity and can be dangerous. Personnel installing and examining specimens must be made aware of this possibility and be protected against injury.

1 Scope

1.1 This part of **ISO 7539** covers procedures for designing, preparing and using bent-beam test specimens for investigating the susceptibility of a metal to stress corrosion.

The term “metal” as used in this of **ISO 7539** includes alloys.

1.2 Bent-beam specimens may be used to test a variety of product forms. They are used principally for sheet, plate or flat extruded material, which conveniently provides flat specimens of rectangular cross-section, but may also be employed for cast material, wire or rod, or for machined specimens of circular cross-section. They can also be used for parts joined by welding.

1.3 Since the preparation of the specimens and the apparatus used for stressing them are both simple and inexpensive, bent-beam specimens are especially suitable for multiple testing and for atmospheric stress corrosion tests.

1.4 Bent-beam specimens are usually tested under nominally constant strain conditions but nominally constant load conditions may be employed. In either case local change of curvature in the specimen when cracking occurs results in changing conditions during crack propagation. The “test stress” is taken as the highest surface tensile stress existing at the start of the test.

2 Normative references The following standards contain provisions which, through reference in this text, constitute provisions of this part of **ISO 7539**. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of **ISO 7539** are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of **IEC** and **ISO** maintain registers of currently valid International Standards.

ISO 7539-1 : 1978, Corrosion of metals and alloys—Stress corrosion testing—Part 1 : General guidance on testing procedures.

ISO 7539-4 : 1989, Corrosion of metals and alloys—Stress corrosion testing—Part 4 : Preparation and use of uniaxially loaded tension specimens.

3 Definitions For the purposes of this part of ISO 7539, the definitions given in ISO 7539-1 are applicable.

4 Principle

4.1 The test consists of applying a bending stress to a beam specimen of rectangular or circular section and exposing the stressed specimen to a specified test environment.

4.2 The magnitude of the resultant applied tensile stress in the outer fibres of the bent-beam specimen is calculated from the dimensions and modulus of elasticity of the specimen and the bending deflection, as described in 5.4.

4.3 Bent-beam specimens are used only for testing at stress levels below the elastic limit since the formulae used for calculating stress in bent beams apply only within the elastic range.

4.4 The time required for cracks to appear after exposure of stressed specimens to the test environment or the threshold stress below which cracks do not appear can be used as a measure of the stress corrosion resistance of the material in the test environment at the stress level employed.

4.5 Wide variations in test results may be obtained for a given metal and environment even when testing nominally identical specimens and the replication of tests is frequently necessary.

4.6 The possibility of relaxation during the exposure period should be considered especially when specimens are exposed at elevated temperatures. Relaxation can be estimated if creep data are available for a simultaneous effect of the test environment. The difference in thermal expansion should also be considered.

5 Specimens

5.1 General

5.1.1 Identification marks or numbers should be permanently inscribed at each end of the specimen. This is the region of lowest stress and the identification marks will therefore not initiate cracking.

5.1.2 Specimens for determination of mechanical properties shall be taken from the same heat treatment batch, and preferably from the same piece of material, as the stress corrosion specimens.

5.2 Types of specimens

5.2.1 Bent-beam stress corrosion specimens are usually flat strips of metal of uniform rectangular cross-section and uniform thickness. They may alternatively be lengths of wire or rod of uniform circular cross-section.

5.2.2 Bent-beam stress corrosion tests may also be carried out on specimens having a gauge length of uniform rectangular or circular cross-section with threaded ends of larger cross-section as described in ISO 7539-4.

5.3 Surface finish

5.3.1 Wire or rod specimens and flat specimens cut from sheet, plate and extruded sections may be tested with the original surface retained. This is often desirable as the structure of the original surface may be different from that of the layers of metal beneath.

5.3.2 If it is desired to exclude the effects of variations in the original surface conditions for a comparison of different alloys, the specimens should be finished by grinding or machining to a depth of at least 0.25 mm. This is usually sufficient to eliminate original surface imperfections without completely removing any outer recrystallized layer. The maximum depth of machining or grinding of the surface should be decided after studying the structure of the material as shown in an etched metallographic section. It is desirable to remove the required amount of metal in several steps by alternatively machining or grinding opposite surfaces. This practice minimizes warping due to unequal residual stresses introduced by machining. All edges should be similarly ground or machined to remove any cold worked material remaining from shearing.

5.3.3 Chemical or electrochemical treatments are generally inappropriate for flat rectangular section specimens as attack at the edges tends to be greater and less easy to control than on the faces.

5.3.4 If chemical or electrochemical treatments are employed, care must be taken to ensure that the conditions used do not result in selective phase attack on the metal or leave a deposit of undesirable residues on the surface.

5.3.5 Chemical or electrochemical treatments that generate hydrogen on the specimen surface must not be used on materials that are susceptible to hydrogen-induced damage.

5.3.6 Before testing, the specimens should be degreased to remove surface contamination; they should then be tested immediately, or stored in such a way as to avoid contamination or deterioration until they can be tested.

5.4 Methods of stressing

5.4.1 Constant strain methods

5.4.1.1 Modes of loading Figure 1 shows six methods of stressing specimens under nominally constant strain conditions. The two-point loaded, three-point loaded and four-point loaded specimens represent the three basic modes of loading used for bent-beam specimens. The double-beam specimen, fully supported specimen and lever-loaded specimen may be regarded as special cases of four-point loading.

5.4.1.2 Two-point loading

5.4.1.2.1 The maximum stress in a two-point loaded specimen occurs at the mid-point of its convex surface and decreases to zero at the specimen ends.