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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 : Guide général des méthodes d'essai

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Foreword

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Corrosion of metals and alloys — Stress corrosion testing —

Part 1 :
General guidance on testing procedures

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 and field of application

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 clause 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 phenonema 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. Attemps 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 in 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^{-1}, 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 for 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^{-1}.

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 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.

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 selecting 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 increased susceptibility of specimens in