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Corrosion of metals and alloys — Stress corrosion testing —

Part 7: Slow strain rate testing

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

Partie 7: Essais à faible vitesse de déformation

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 7539-7 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys.*

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

- *Part 1: General guidance on testing procedures*
- *Part 2: Preparation and use of bent-beam specimens*
- *Part 3: Preparation and use of U-bend specimens*
- *Part 4: Preparation and use of uniaxially loaded tension specimens*
- *Part 5: Preparation and use of C-ring specimens*
- *Part 6: Preparation and use of pre-cracked specimens*
- *Part 7: Slow strain rate testing*
- *Part 8: Preparation and use of welded specimens*

Annex A of this part of ISO 7539 is for information only.

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Introduction

This part of ISO 7539 is one of a series giving procedures for designing, preparing and using various forms of test specimen to carry out tests to establish a metals resistance to stress corrosion.

Each of the standards in the series needs to be read in association with ISO 7539-1. This helps in the choice of an appropriate test procedure to suit particular circumstances as well as giving guidance towards assessing the significance of the results of the tests.

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Corrosion of metals and alloys — Stress corrosion testing —

Part 7: Slow strain rate testing

1 Scope

1.1 This part of ISO 7539 covers procedures for conducting slow strain rate tests for investigating susceptibility of a metal to stress corrosion cracking, including hydrogen-induced failure.

The term "metal" as used in this part of ISO 7539 includes alloys.

1.2 Slow strain rate tests are adaptable for testing a wide variety of product forms, including plate, rod, wire, sheet and tubes, as well as composites of these and parts joined by welding. Notched or pre-cracked specimens may be used, as well as initially plain specimens.

1.3 The principal advantage of the test is the rapidity with which susceptibility to stress corrosion cracking of a particular metal/environment combination can be assessed.

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 listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 7539-1 : 1987, *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.*

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

3 Definitions

For the purposes of this part of ISO 7539, the following definitions and those given in ISO 7539-1 apply.

3.1 creep: Time-dependent mechanical deformation of a specimen after application of the initial load.

3.2 elongation to fracture: The ratio, expressed as a percentage, of the increase in length of the gauge length which has occurred during a test, to the original gauge length.

3.3 maximum load: The maximum value of the load achieved during a test taken to total failure or, in the case of composite materials, the load corresponding to failure of one element.

3.4 nominal stress — elongation curves: A plot of the nominal stress calculated from the instantaneous applied load and the original cross-sectional area of a specimen, against the elongation of the gauge length at the time of the load measurement.

3.5 reduction of area: The ratio, expressed as a percentage, of the maximum decrease in cross-sectional area which has occurred during a test, to the original cross-sectional area.

3.6 strain rate: The initial rate of increase of the gauge length of an initially plain tensile specimen.

4 Principle

4.1 The test consists in subjecting a specimen to an increasing strain whilst exposed to a specified environment with a view to determining stress corrosion susceptibility by reference to one or more of the parameters enumerated in clause 7.

4.2 Corrosive environments may cause a deterioration of the properties of stressed materials beyond those observed with the same combination of environment and material when the latter is not subjected to slow dynamic strain. This enhanced deterioration, usually due to the initiation and growth of cracks, may be expressed in a number of different ways for the purpose of assessing stress corrosion susceptibility.

4.3 Tests may be conducted in tension or in bending, on initially plain, notched or pre-cracked specimens. The most important characteristic of the test is the relatively slow strain rate generated at the region of crack initiation or growth in the metal, hence the preference for such tests being referred to as slow strain rate tests.

5 Specimens

5.1 A variety of specimen shapes and sizes can be used, but those most commonly employed are described in ISO 7539-4 and ISO 7539-6.

5.2 The remarks in the aforementioned documents on specimen design, preparation and gripping are equally applicable to specimens for slow strain rate tests.

6 Procedure

6.1 The equipment required for slow strain rate testing is a device that permits a selection of deflection rates whilst being powerful enough to cope with the loads generated. Deflection rates that have been used most frequently in testing initially plain specimens are in the range $10^{-3}\ s^{-1}$ to $10^{-7}\ s^{-1}$.

6.2 Notched or pre-cracked specimens may be used when it is desired to restrict cracking to a particular location, for example when testing the heat-affected zone associated with a weld or whenever a given piece of material exhibits a range of mechanical properties that would be likely to promote different strain rates in different parts of a specimen. Notched or pre-cracked specimens may also be used to restrict load requirements, where bending, as opposed to tensile loading, may offer further advantages.

6.3 For initially plain specimens, especially with a waisted gauge length, the strain rate at the outset of the test is readily defined, but once cracks are initiated and have grown to some extent in such specimens, straining is likely to be concentrated in the material in the vicinity of the crack tip and may not be the same as the initial strain rate. Rigorous solutions for the strain rate at crack tips or notches are not yet available, but it is likely that the effective strain rates will be higher than those for the same deflection rates applied to plain specimens.

6.4 Tests may involve taking a specimen to total failure, and assessing the mode of failure to determine susceptibility to stress corrosion cracking, or stopping a test at some intermediate stage and then determining the extent of crack initiation or growth.

6.5 Experience suggests that for initially plain specimens tested in tension a strain rate in the region of $10^{-6}\ s^{-1}$ will be appropriate for the initial test. The absence of stress corrosion cracking from such a test is not necessarily indicative of immunity from stress corrosion cracking in the system studied, since susceptibility is known to be a function of, amongst other parameters, strain rate (see annex A). Subsequent tests at other strain rates, such as $10^{-5}\ s^{-1}$ and $10^{-7}\ s^{-1}$, should be conducted if the initial test produces no evidence of stress corrosion cracking. If specimens are pre-loaded to reduce the total test time during tests at very slow strain rates, the comments made in 7.4.3 may be important.

6.6 The establishment of cracking conditions in a given metal/environment combination may be time-dependent, if they do not exist at the outset of the test. In such circumstances stress corrosion cracking may only be observed if the strain rate is sufficiently slow to ensure that overload failure does not occur before the necessary time has elapsed whereby the necessary environmental conditions for cracking have been established. These difficulties can sometimes be avoided by exposure of the specimens to the test environment for some time prior to the initiation of dynamic strain.

6.7 The environmental testing conditions selected depend upon the purpose of the test but, ideally, should be the same as those prevailing for the intended use of the metal or comparable to the anticipated service condition. In practice, a number of standard environments is used for ranking purposes, but application of the results obtained for predicting service behaviour depends on an understanding of the system or on correlation with experience.

6.8 It is recommended that wherever possible the gripped portions should be excluded from contact with the corrosive environment. If this is not possible, the problems that may arise include the following:

a) galvanic effects will almost invariably influence results if the grips are made from a material different from that of the test piece and electrical insulation is then necessary;

b) crevice corrosion may occur within the confines of the restricted spaces between grips and test pieces and stress discontinuities can lead to premature stress corrosion failure in such regions;

c) crevice problems may arise also where the test piece emerges from the test cell and these should be avoided by appropriate design of the cell, by the use of protective coatings at such positions or by enlargement of the cross-sectional area of the test piece beyond the parallel portion.

6.9 Where the test is simply used to determine whether or not stress corrosion cracking occurs, it is recommended that straining of the specimens should be started after being brought into contact with the environment.

6.10 Where specimens are taken to the point of total failure in slow strain rate tests, it is recommended that specimens should be tested in an inert environment as well as in the corrosive test environment at the same temperature and at the same rate. This permits a comparative assessment of the effects of the corrosive environment by providing baseline data relating to inert conditions. For some materials, including high strength aluminium alloys and steels, it may not be sufficient to assume that a test in air constitutes a test in an inert environment.

6.11 It is recommended that specimens without applied straining should be exposed to the same conditions as strained specimens. Metals may suffer deterioration in mechanical properties by contact with corrosive environments even in the absence of applied strain (e.g. pitting, intergranular corrosion, etc.) and the effect of applied straining can only be assessed by comparison with the behaviour of unstrained specimens.

6.12 Temperature variations during tests, particularly at very low strain rates and high temperatures, can themselves modify the strain rate and should be avoided if they significantly influence results.

7 Assessment of results

7.1 Where specimens are taken to total failure, evidence of stress corrosion cracking is usually apparent from visual examination by low power microscopy for secondary cracking or by change in the failure mode as shown by fractographic assessment of the fracture surface.

7.2 Average stress corrosion crack velocities may be determined from the length of the longest crack measured on the fracture surfaces of specimens that have failed completely or on sections through specimens that have not proceeded to total failure, divided by the time of testing. Although this parameter assumes that cracking is initiated at the start of the test, which is not always the case, nevertheless such a measurement is frequently found to be in reasonable agreement with those made more precisely. With pre-cracked specimens other methods are available for monitoring crack growth (see ISO 7539-6) whereby crack velocities may be determined.

7.3 Comparison between identical specimens exposed to the test environment and to an inert environment may be used for assessing the susceptibility to stress corrosion cracking. Increasing susceptibility to cracking is indicated by increasing departure from unity of the ratio

$$\frac{\text{results from specimen in test enviroment}}{\text{results from specimen in inert environment}}$$

applied to one or more of the following parameters of the same initial strain rate:

a) time to failure;

b) ductility, assessed by e.g. reduction in area or elongation to fracture;

c) maximum load achieved;

d) area bounded by nominal stress/elongation curve;

e) percentage of stress corrosion cracking on the fracture surface.

7.4 Slow strain rate tests can also be used for determining the threshold stress above which detectable cracking occurs at a given strain rate. In some systems, the threshold is likely to be a function of strain rate. Therefore tests should be conducted over an appropriate range of strain rates for the system under consideration in order to ensure that a conservative value is obtained. The procedure to be adopted involves subjecting a number of specimens to different stress ranges following the schedule outlined in 7.4.1 to 7.4.4 for each strain rate.

7.4.1 The first specimen is taken to total failure and the average stress corrosion crack velocity is determined using the procedure outlined in 7.2.

7.4.2 The minimum time for the development of a detectable crack at stresses in excess of the threshold value is then estimated by dividing the minimum crack length that can be readily distinguished from other surface imperfections and measured accurately during metallographic examination at a magnification of about $\times$ 500 by the average stress corrosion crack velocity. This time is employed as the test duration for the remaining tests in order to minimize the stress change during each test. The stress range is defined as that which occurs during the application of slow straining.

7.4.3 The second specimen is loaded at a relatively rapid rate in conditions where stress corrosion cracking cannot occur, until an appropriate initial stress level, for example 50 % of the tensile strength observed in the first test, is attained. The displacement is then held constant until any time-dependent deformation has diminished to a rate considered to be below that of the strain rate to be applied subsequently. This period may be considerable, of the order of days, in some systems. The environmental conditions for cracking are then established and slow straining is subsequently initiated. The test is discontinued after the appropriate duration as given in 7.4.2. The specimen is examined for cracks, preferably by microscopy of an axial section, so that an average crack velocity can be determined.

7.4.4 Further tests are conducted likewise using other initial stresses in accordance, for example with the binary search procedure given in ISO 7539-1, until a threshold value is defined below which no evidence of cracking is found and above which the average crack velocity is significant. A plot of stress range for each test against average crack velocity can be helpful in defining a threshold.

8 Test report

The test report should include the following information:

a) full description of the test material from which the specimens were taken, including composition and structural condition, type of product and section thickness;

b) orientation, type and size of test specimens and their surface preparation;

c) straining procedure including initial strain rate for plain specimens and deflection or COD rate for pre-cracked or notched specimens;

d) test environment, including electrode potential and/or current density, temperature, pressure, etc. where appropriate;

e) methods used in defining test results (time to total failure, number and location of cracks, average crack velocity, remnant strength and ductility, percentage of stress corrosion cracking on fracture surface).

Annex A
(informative)

Strain rate
(see 6.5)

It is probable that the fastest strain rate that will promote stress corrosion in a given system depends upon the stress corrosion crack velocity. In general, the lower the stress corrosion crack velocity the slower the initial strain rate needed to promote cracking. Initial strain rates that have promoted cracking in certain systems are given in table A.1.

Table A.1

System	Initial strain rate s^{-1}
Aluminium alloys in chloride solutions	10^{-6}
Copper alloys in ammoniacal solutions	10^{-6}
Ferritic steels in carbonate, hydroxide or nitrate solutions	10^{-6}
Magnesium alloys in chromate/chloride solutions	10^{-5}
Nickel based alloys in high temperature water	10^{-7}
Stainless steels in chloride solutions	10^{-6}
Stainless steels in pure water	10^{-6}
Titanium alloys in chloride solutions	10^{-5}

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