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Corrosion of metals and alloys — Stress corrosion testing —

Part 7:
Method for slow strain rate testing

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

Partie 7: Méthode d'essai à faible vitesse de déformation

Reference number
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Foreword

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

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

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

This second edition cancels and replaces the first edition (ISO 7539-7:1989), Clauses 1, 3, 4, 6, 7 and 8 of which have been technically revised.

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

- *Part 1: General guidance on testing procedures*
- *Part 2: Preparation and use of bent-beam specimens*
- *Part 3: Preparation and use of U-bend specimens*
- *Part 4: Preparation and use of uniaxially loaded tension specimens*
- *Part 5: Preparation and use of C-ring specimens*
- *Part 6: Preparation and use of pre-cracked specimens for tests under constant load or constant displacement*
- *Part 7: Method for slow strain rate testing*
- *Part 8: Preparation and use of specimens to evaluate weldments*
- *Part 9: Preparation and use of pre-cracked specimens for tests under rising load or rising displacement*

Corrosion of metals and alloys — Stress corrosion testing —

Part 7:
Method for slow strain rate testing

1 Scope

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.

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 specimens may be used, as well as initially plain specimens.

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 referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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 7539-6:2003, *Corrosion of metal and alloys — Stress corrosion testing — Part 6: Preparation and use of pre-cracked specimens for tests under constant load or constant displacement*

3 Terms and definitions

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

3.1
creep
time-dependent mechanical deformation of a specimen after application of the initial load

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

3.3

maximum load

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

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

plastic strain to failure

estimated plastic contribution to the total strain to failure determined by subtracting the elastic strain at failure from the total strain at failure

3.6

reduction of area

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

3.7

strain rate

initial rate of increase in gauge length of an initially plain tensile specimen

4 Principle

4.1 The test consists of subjecting a specimen to 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 or notched 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 utilized are described in ISO 7539-4 and ISO 7539-6.

5.2 The remarks in the aforementioned documents concerning 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 consists of 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 specimens may be used when it is desired to restrict cracking to a particular location, e.g. 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 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 notches are not yet available, but it is likely that the effective strain rates will be higher than those for the same displacement rates applied to plain specimens.

6.4 Tests may involve taking a specimen to total failure, and assessing the mode of failure in order 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 $10^{-6}\ s^{-1}$ is 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.

6.6 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.

6.7 Environmental factors of importance are electrode potential, temperature, solution composition, pH, concentration of dissolved gases, flow rate and pressure. ISO 7539-1 provides useful background information. In relation to gaseous environments a critical factor is purity of the gas.

6.8 Tests may be conducted under open circuit conditions in which the electrode potential of the metal is dependent on the specific environmental conditions of the test, of which the degree of aeration is an important factor. Alternatively, the electrode potential may be displaced from the open circuit value by potentiostatic or galvanostatic methods.

6.9 Auxiliary electrodes to apply external current should be designed to produce uniform current distribution on the specimen, i.e. the electrode potential should be constant.

6.10 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.

6.11 It is recommended that wherever possible the gripped portions 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.

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

6.13 Where specimens are taken to the point of total failure in slow strain rate tests, it is recommended that specimens 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.

6.14 It is recommended that specimens without applied straining 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.

6.15 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 a 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 depth of the deepest 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 notched 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:

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

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

- time to failure;
- plastic strain to failure;
- ductility, assessed by, e.g., reduction in area or elongation to fracture;
- maximum load achieved;
- area bounded by nominal stress/elongation curve;
- percentage of stress corrosion cracking on the fracture surface.

It should be recognized that in most testing, the displacement of the gauge section is not measured directly. Rather, the crosshead displacement is measured and this includes a contribution from the displacement of the shoulders of the specimen and of the load train. Since these can vary from one test system to another, the calculated strain on the gauge section of the material at any time will be sensitive to the test system. Accordingly, the actual strain rate on the gauge section in the elastic loading region will also vary from one test system to another despite similar values of the nominal strain rate. If failure occurs in the elastic region, a correction shall be made by initially determining the relationship between the displacement on the gauge length and the crosshead displacement; e.g. by measuring the displacement of the gauge length directly in a prior air test. This “calibration” data would be used to set the strain rate for the test and to calculate the elastic strain to failure on the gauge length.

However, when yielding occur, most of the displacement in the crosshead is associated with the plastic deformation of the gauge section and the differences between test system should be very much less significant. Accordingly, for those systems which fail above yield, meaningful comparison of data can be made by use of the plastic strain to failure, E_p, defined by:

$$E_\mathrm{p}\,(\%) = \left\{ \frac{XT_\mathrm{F}}{L_1} - \left[\frac{\sigma_\mathrm{F}}{\sigma_\mathrm{PL}} \right] \times \frac{XT_\mathrm{PL}}{L_1} \right\} \times 100$$

where

X is the extension rate in metres per second;

T_F is the time to failure in seconds;

L_1 is the initial gauge length in metres;

T_PL is the time to the proportional limit in seconds;

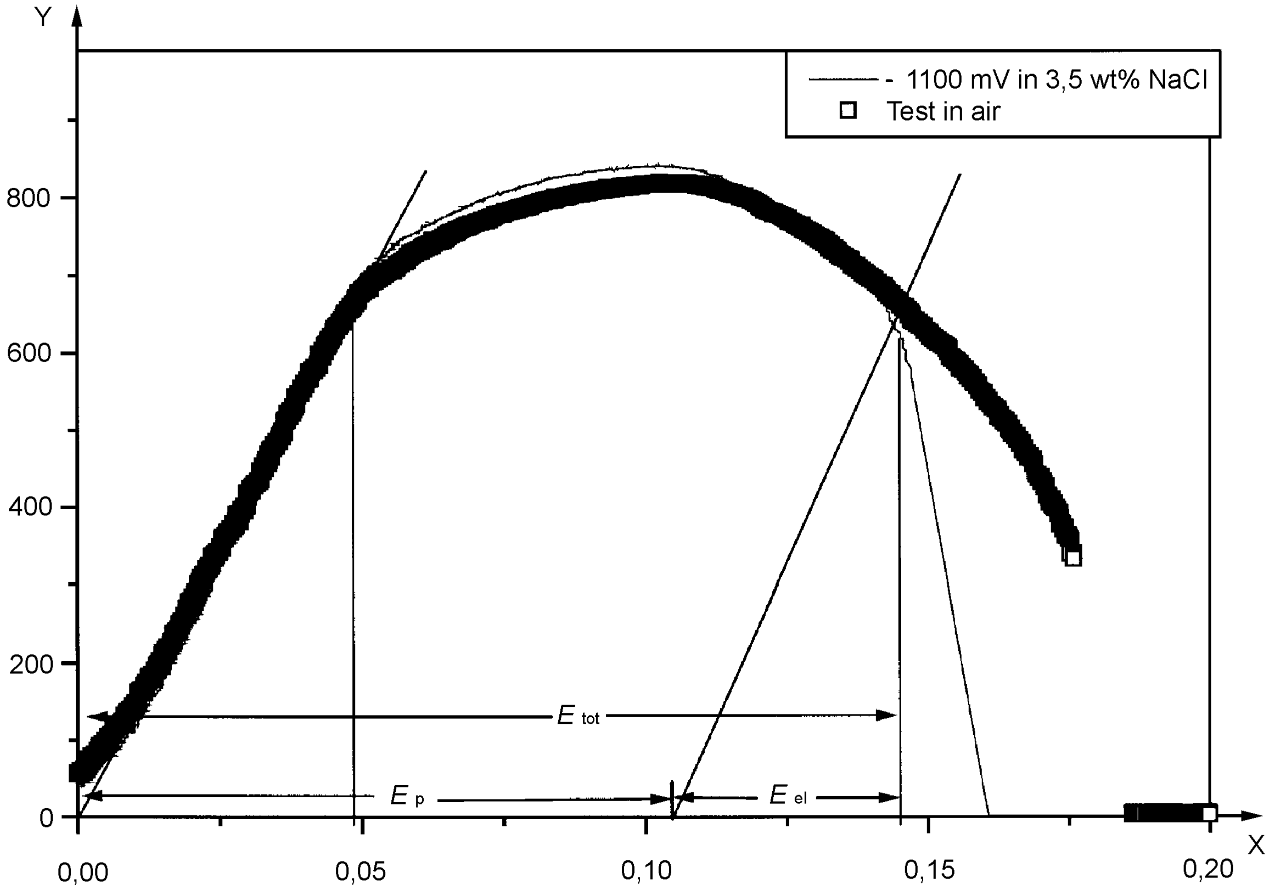
σ_F is the stress at failure;

σ_PL is the stress at proportional limit.

If significant work hardening occurs, this definition of the plastic strain to failure is not ideal, because of the additional elastic contribution including that from the specimen shoulder and load train. The strain rate may also be test-system-sensitive in this regime also. Nevertheless, the definition of E_p shall be used but its limitations recognized. See Figure 1.

7.4 The interrupted slow strain rate test can be used for estimating approximately the threshold stress or threshold strain 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. In testing with very low strain rates, e.g. 10^{-8} s^{-1}, a higher strain rate may be adopted during elastic loading of the specimen.

The interrupted test involves stopping the test at a specific strain or stress level, removing the specimen, and inspecting the surface for cracks at a magnification of × 500. If a crack is observed, the test is repeated with a fresh specimen but with the stress or strain value at the interrupt point lowered. Conversely, for the situation when no crack is observed when first stopping the test, the test is repeated with a higher value for the interrupt stress or strain. Further tests are conducted likewise until a value of the threshold stress or strain is obtained at which no crack is observed but above which cracking is seen. The magnitude of the changes in the stress or strain at which successive tests are interrupted will determine the range of uncertainty of the threshold stress or strain. The principles embodied in the binary search procedure given in ISO 7539-1 may be applied in order to facilitate determination of the threshold value.



Key

X strain

Y stress in megapascals

Figure 1 — Illustration based on data for a super 13 Cr stainless steel showing basis for determining the plastic strain to failure, E_p, where E_{tot} is the total elongation and E_{el} is the elastic elongation

8 Test report

The test report shall 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 displacement rate for notched specimens;

d) starting procedure for the test including, if applicable, initial stress level and interval prior to the commencement of slow straining;

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

f) methods used in defining test results (e.g. time to total failure, elongation to fracture, reduction in area, plastic strain to 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 highest 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 — Initial strain rates that have promoted cracking in various systems

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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