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Corrosion of metals and alloys — Evaluation of stress corrosion cracking by the drop evaporation test

Corrosion des métaux et alliages — Évaluation de la résistance à la fissuration par corrosion sous contrainte par essai d'évaporation goutte à goutte

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15324 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

Annexes A, B and C of this International Standard are for information only.

Corrosion of metals and alloys — Evaluation of stress corrosion cracking by the drop evaporation test

1 Scope

1.1 This International Standard specifies the procedure for determining the relative resistance of stainless steels and nickel-base alloys to stress corrosion cracking in a sodium chloride drop evaporation system.

1.2 The method results in a threshold stress to fracture, the magnitude of which can be used to rank the relative performance of different alloys for this environment.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

ISO 4287:1997, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*.

ISO 4288:1996, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Rules and procedures for the assessment of surface texture*.

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

3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

3.1
threshold stress
stress below which no fracture in 500 h is observed (see also 8.8)

3.2
time-to-fracture
elapsed time from start of test until fracture of the specimen

4 Principle

4.1 A dilute salt solution is dripped on to a heated tensile specimen of the material under test which is held horizontally and subjected to a uniaxial load.

4.2 Tests are carried out at different applied stresses and the time to specimen fracture is recorded. A threshold stress is defined based on the principle of no observed fracture in 500 h.

4.3 The elevated temperatures, wetting and drying process and associated concentration of salt with the drop evaporation process represent a potentially severe environmental condition which could induce stress corrosion cracking in some alloys. As such, it represents a severe test for alloy performance.

4.4 Evaporation of salt solutions leading to salt concentration at elevated temperature is not uncommon in service and can arise, e.g., in sea water spray situations or as a result of solution leaks dropping on to hot surfaces. However, the variability of service conditions means that this method is suitable only as a method of ranking performance, based on the magnitude of the threshold stress, and not as an acceptance test for service application.

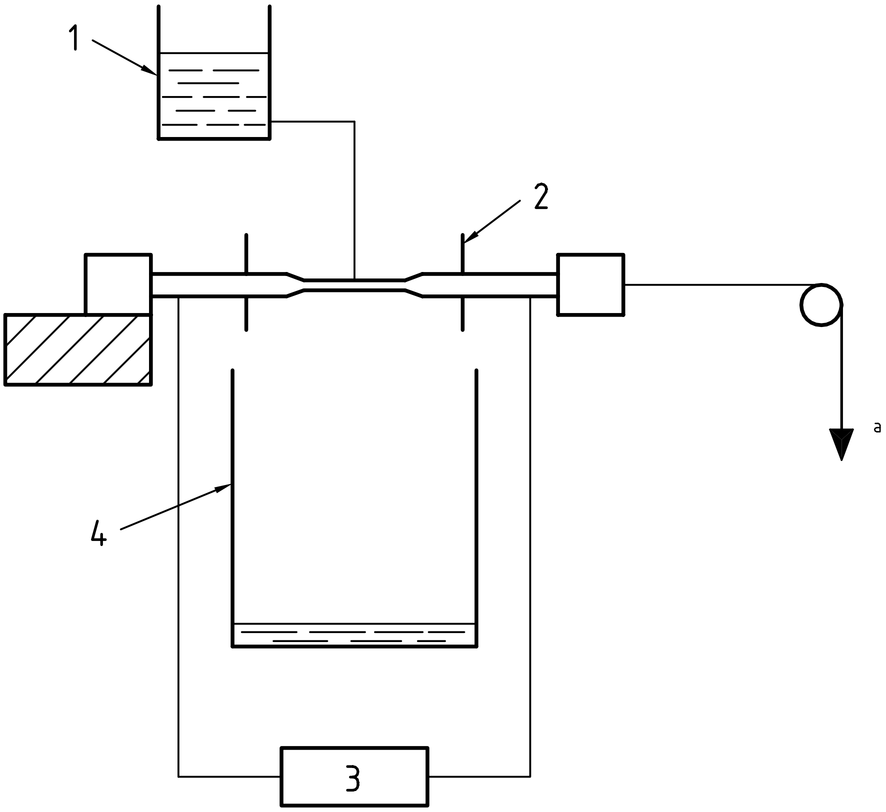
5 Apparatus

5.1 System for applying a constant load to a specimen held in a horizontal configuration.

5.2 Equipment for applying an alternating or direct current of up to about thirty amperes current to the specimen at a voltage of two volts or less.

NOTE The current required will depend on the dimensions of the specimen and the properties of the material.

5.3 A reservoir, (typically 20 l) for the sodium chloride solution connected to a vertical glass tube with internal and external diameters approximately 0,4 mm and 4 mm respectively. The bottom of the glass tube is held at a distance of approximately 10 mm above the centre of the specimen (see Figure 1).



Key

1	0,1 mol/l sodium chloride
2	Plastic shield to protect electrical connections
3	A.C. or D.C. electrical supply
4	Vessel to collect solution

[a] Direction of applied load (force).

Figure 1 — Schematic arrangement for drop evaporation tests

The system shall be capable of delivering a controlled flow of solution of 10 drops/min $(\pm 10\,\%)$ (see annex A). The composition of the solution in the reservoir shall be maintained constant.

5.4 A suitable calibrated device for measuring the surface temperature of the specimen. Surface temperature measurement is important because of the need to ensure that the method of measurement itself does not affect the temperature. Applicable methods are described in annex B.

5.5 A device for detecting specimen fracture and recording the time to fracture. A microswitch attached to the specimen mounting and associated with an electric timer is suitable.

5.6 Some degree of containment of the test system, to limit the possibility of fluctuations in temperature due to air currents, as necessary.

6 Test specimen

6.1 The specimens shall be tensile specimens with a circular cross section in the gauge length.

6.2 The specimens shall be prepared from thick sheet, plate, bar, strip or tube.

6.3 The specimens shall be cut such that the longitudinal direction of the specimen is parallel to the rolling direction of the original processed material.

6.4 The specimen shall have a gauge length of 10 mm and a gauge diameter of 1,8 mm. The gauge length should be faired into the grip section with suitable gradual radii. The dimensions of the main body of the specimen affect heat transfer and the currents required.

NOTE A diameter of 5 mm or a 2 mm square cross section is effective. The overall length of the specimen should be about 100 mm.

6.5 The specimen shall be produced to a fine ground finish ($Ra < 1\,\mu$m) preferably by grinding in the longitudinal direction as any grinding flaws are then parallel to the stress axis. The surface texture can be measured in accordance with ISO 4287 or ISO 4288 by conventional stylus or laser profilometry. The texture should be measured along the longitudinal axis and the process repeated a number of times (typically about six) after rotating the specimen. It should be emphasised that the Ra value, although most commonly quoted is not necessarily the critical parameter with respect to stress corrosion cracking and can be useful in evaluating and reporting other measures of surface texture as discussed in annex C.

NOTE Residual stresses and strain-hardened layers can have an influence on stress corrosion cracking. The effect can be significant when test specimens are removed from material in which complete stress relief is impractical, such as weldments, as-quenched materials and complex forged or extruded shapes. In addition, near-surface residual stresses can be introduced during machining and surface grinding and these processes should be undertaken in such a way as to minimize the generation of residual stress. In some cases, relief of surface residual stress may be undertaken provided that this does not lead to a change in microstructure. The surface cold worked layer induced by the grinding process may also be removed by electrolytic polishing. Measurement of residual stress is desirable.

6.6 After surface finishing, surface contaminants shall be removed by an appropriate cleaning procedure.

NOTE Rinsing with distilled water, followed by alcohol and a non-chlorinated solvent is adequate for most cases. The use of an ultrasonic bath is recommended.

7 Test solution

The test solution is 0,1 mol/l NaCl, prepared from analytical reagent grade chemicals and distilled or deionised water of conductivity $\leqslant 10\,\mu$S cm^{-1}, see ISO 3696. The solution in the reservoir shall be maintained at a temperature of (23 ± 3) °C. The initial pH of the solution shall be in the range 5,7 to 7,0 in this temperature range.

NOTE By agreement, the method can be used to evaluate performance in other solutions, e.g. sea water. The use of sea water results in formation of a more acidic salt during drying and will tend to be more aggressive than 0,1 mol/l NaCl.

8 Procedure

8.1 The specimen is attached to the gripping devices.

8.2 The specimen is resistance heated until a steady temperature of 300 °C is attained. The current required to maintain this temperature is noted.

NOTE The method described in annex B can be used to define the current required to achieve the desired temperature. However, it is recommended that the surface temperature be measured in each test using a small contact thermometer or thermocouple calibrated as described in annex B.

8.3 The solution is dripped on to the specimen at a rate of 10 drops/min ± 10 %.

8.4 The load is then applied. The load to be applied shall be a fraction of the load corresponding to the 0,2 % proof stress at a temperature of 100 °C. A binary search procedure may be employed to determine the threshold stress as described in ISO 7539-1, although judicious selection of the stress for the first test should be used based on the anticipated resistance of the alloy.

NOTE Because of the dependence of yield stress on temperature, application of the load prior to dripping of the solution would not be appropriate as a general rule because it may result in exceeding the yield stress of the specimen.

8.5 The specimen fracture detection system is activated.

8.6 The applied current is increased by 25 %. This gives rise to a situation in which the specimen just dries out between each drop.

NOTE The application of the final current prior to wetting will cause the temperature to increase to an unacceptable level in terms of material property change, but such a current increase is necessary to establish test conditions severe enough to cause fracture in a reasonable timescale.

8.7 The temperature of the specimen shall be measured in the dry region at least once during the test, see annex B.

8.8 The test is continued until the specimen fractures, up to a maximum of 500 h. The time to fracture is recorded.

NOTE By agreement, the maximum exposure time can be increased and the threshold stress redefined accordingly. Nevertheless, the value corresponding to 500 h of testing should be quoted.

8.9 The fractured specimen shall be examined to confirm that the failure was due to stress corrosion cracking, e.g. by fractographic or metallographic methods.

8.10 The test is repeated at other fractions of the yield strength until the minimum stress to cause fracture in 500 h is determined. The value shall be that fraction of the yield strength at which fracture did occur, but which was just above the stress at which no fracture occurred in 500 h.

9 Test report

The test report shall include the following information:

a) full description of the test material from which the specimens were taken, including UNS number, composition, heat treatment, type of product, and the mechanical properties;

b) method of manufacture of the specimens, details of the surface preparation and surface roughness parameters;

c) the solution composition, pH, volume and temperature;

d) the temperature of the specimen, quoting maximum, minimum and mean values and quoting the method of measurement;

e) the applied stresses as a fraction of the yield stress at 100 °C together with the value of the yield stress at 100 °C;

f) the visual appearance of the failed specimens (at a magnification of × 20) at the end of the test together with the results of the metallographic examination;

g) the time-to-fracture for each applied stress plus the value of the estimated threshold stress to fracture.

Annex A
(informative)

Drop rate control

A pump can be used to deliver the solution directly to the glass tube (see 5.3) provided it maintains constancy of rate of supply. When using peristaltic pumps, variation of the flow rate at discrete intervals has been observed when the roller of the pump “lifts” off the silicon rubber tubing. This can cause a periodic temperature excursion.

Suitable control of drop rate can be obtained by direct gravity feed by passing the solution through a length (about 250 cm) of 0,5 mm internal diameter polytetrafluoroethylene capillary tubing connected between the reservoir and the glass tube, which is typically about 5 cm in length. The drop rate is adjusted by raising or lowering the reservoir or solution level. This may be necessary in maintaining constancy of drop rate as the solution level decreases with time.

Annex B
(informative)

Surface temperature measurement

A wax indicator stick which melts at the critical temperature can be used to measure the temperature in a control sample for the purpose of determining the required current. Paints which change colour with temperature may also be used.

If a contact thermometer is used the indicated surface temperature will be in error; the actual surface temperature will be much higher. For example, a 0,5 mm stainless steel clad thermocouple, resting on a specimen and indicating a temperature of 300 °C can underestimate the actual temperature by 200 °C. Nevertheless, the contact thermometer can be calibrated approximately using the measurement from waxes or paints. Bulky surface temperature probes can lower the temperature markedly when in position and are not recommended.

There is no ideal method of measuring the temperature during dripping of the solution. Optical methods can be used but are uncertain due to the changing emissivity and small size of the sample in its gauge length. The use of a contact thermometer at these lower temperatures is satisfactory for giving an indication when applied for short intervals but can still produce errors, the extent of which will depend on the location of the probe. Each drop of solution normally flows along the specimen on to one of the shoulders of the specimen. The presence of a probe can retain the drop in the centre of the gauge length and thus greatly reduce the temperature whilst the probe is in position. If measurements are made in the wet zone, the presence of the probe, even for a short period, can cause the behaviour of the solution drops to change and markedly alter the surface temperature. Nevertheless, the temperature of the specimen at the dry edge of the specimen can be monitored with a calibrated probe.

Annex C
(informative)

Surface texture parameters — relevance to stress corrosion cracking

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

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

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

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