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Corrosion of metals and alloys — Stress corrosion testing —

Part 12:
Requirements for atmospheric stress corrosion cracking testing

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

Partie 12: Exigences relatives aux essais de fissuration par corrosion sous contrainte atmosphérique

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Foreword

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This document was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

A list of all parts in the ISO 7539 series can be found on the ISO website.

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Corrosion of metals and alloys — Stress corrosion testing —

Part 12:
Requirements for atmospheric stress corrosion cracking testing

1 Scope

This document specifies the general requirements for designing and conducting tests to assess the susceptibility of metals and alloys to stress corrosion cracking under atmospheric exposure conditions.

The testing methodology includes exposure to natural environments and environments in artificially accelerated laboratory tests.

Applications include aluminium and its alloys in aerospace and construction; stainless steels in construction, pressure vessels, and nuclear waste containment; high strength low alloy steels in automotive, construction, and in lifting chains.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 6892-1, *Metallic materials — Tensile testing — Part 1: Method of test at room temperature*

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

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

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

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

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

ISO 7539-6, *Corrosion of metals and alloys — Stress corrosion testing — Part 6: Preparation and use of precracked specimens for tests under constant load or constant displacement*

ISO 7539-8, *Corrosion of metals and alloys — Stress corrosion testing — Part 8: Preparation and use of specimens to evaluate weldments*

ISO 8044, *Corrosion of metals and alloys — Vocabulary*

ISO 8565:2011, *Metals and alloys — Atmospheric corrosion testing — General requirements*

ISO 9225, *Corrosion of metals and alloys — Corrosivity of atmospheres — Measurement of environmental parameters affecting corrosivity of atmospheres*

ASTM F21, *Standard test method for hydrophobic films by the atomizer test*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 7539-1, ISO 7539-6, ISO 7539-8, ISO 8044, and the following apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

3.1

atmospheric stress corrosion cracking
environmentally induced cracking due to the exposure of metals and alloys under nominally static stress to the atmosphere, usually accompanied with deposition of salts and pollutants

4 Principle

Atmospheric corrosion depends on the quantity of water in the film formed on the metal surface, which varies as the metal surface wets and dries, and the nature and extent of deposition of particles, aerosols, and gaseous pollutants and contaminants. The corrosivity of the environment changes during the wetting and drying cycle. On initial wetting, the corrosivity increases as the accumulated pollutants on the surface dissolve. As the surface wets further, the solution dilutes and becomes less corrosive. A subsequent decrease in the relative humidity causes the surface water content to decrease. The corrosivity of the solution then increases because of concentration of salts. Oxygen diffusion to the reacting surface is also enhanced during the initial wetting and the later drying process as the diffusion layer thickness is smaller under these conditions. Correspondingly, the susceptibility of the metal to localised corrosion and stress corrosion cracking is enhanced during the transition from wet-to-dry and vice versa.

The primary challenge in atmospheric stress corrosion cracking testing is how best to account for the time variation of the environment (i.e. temperature, humidity, gaseous pollutants etc.), which can change on a very irregular basis, superimposed on daily and seasonal cycles. Furthermore, there can be features of the local environment such that the chemistry of the species forming on the surface can be complex and not characterised by broad classifications such as marine and industrial.

Testing in a representative natural environment would appear the most appropriate way of accounting for realistic exposure conditions, whether outdoors or in some form of construction. However, adopting such an approach can be constrained by the required long-term nature of exposure, to allow for the progressive build-up of deposits and to ensure statistical validity of the data. Information on temperature and wetting and drying cycles can also be incomplete, and there are challenges for in situ crack growth monitoring.

Laboratory testing offers the opportunity for accelerated testing in artificial atmospheres, commonly by application of salt deposits from the outset that can be more aggressive, and by selecting temperature and humidity conditions that can be more conducive to stress corrosion cracking. The challenge arises in using laboratory data for predicting behaviour under practical exposure conditions.

Existing standards for atmospheric corrosion do not include stress corrosion cracking testing, and stress corrosion cracking testing standards do not adequately account for atmospheric exposure testing. This document aims to appropriately specify best practice in combining both features.

5 Test specimen preparation

5.1 The stress-strain response of the alloy shall be determined prior to any stress corrosion cracking test and the proof stress, ultimate tensile stress and elongation to fracture shall be quantified in accordance with ISO 6892-1.

5.2 The test specimen shall be manufactured as specified in the appropriate test standard: ISO 7539-2, ISO 7539-3, ISO 7539-4, ISO 7539-5, ISO 7539-6 or ISO 7539-8. Other specimen geometries can be considered depending on material dimensions, crack orientation, or the application of coatings. [1]

NOTE In most applications, the long-term nature of testing usually necessitates the use of self-contained loading and specimen assemblies.

5.3 A range of possible specimen configurations including self-loaded 2-, 3-, or 4-point bend, C-ring, or tensile test specimens may be adopted. U-bend specimens are commonly used because of their simplicity and cheapness, and notched fracture mechanics specimens such as double cantilever beam are also adopted.

NOTE For testing under controlled salt deposition conditions in a laboratory, flat test specimens stressed in 2- , 3-, or 4-point bend or in tension can be preferable to optimise uniformity of salt deposition and to minimise gravity-induced flow of corrosion products.

5.4 The surface shall be prepared to a well-defined finish (e.g. ground, polished, electropolished) and the average surface roughness, *Ra*, measured.

5.5 For some alloys, a machined and ground finish can result in significant changes in near-surface microstructure and hardness, and introduce residual stress and physical defects. Characterisation of the near-surface microstructure should be undertaken. Measurement of near-surface residual stress should also be performed unless test specimens are loaded significantly beyond yield (e.g. U-bend), since relaxation would ensue in that case.

5.6 The surface shall be degreased with a suitable degreasing agent and the efficacy of this process demonstrated using an atomizer test method, as specified in ASTM F21.

6 Application of stress

6.1 The stress should be applied in accordance with the particular test standard adopted.

6.2 Fixed displacement testing can result in stress relaxation due to short-term and long-term creep, which also varies with temperature, to crack development, and/or material loss. When the goal is to load the specimen to a specified stress, such as in a self-loaded tensile test specimen using a proof ring or similar device, compensation for short-term relaxation can be made by re-loading the specimen to the desired stress and checking the extent of stress relaxation. Typically, the reduction in stress should be less than 1 % of the set value over a 12 h period at ambient condition. For such test specimens, the stress shall be determined upon test conclusion to evaluate the extent of stress relaxation over the whole exposure period.

7 Natural exposure testing

7.1 The atmospheric corrosion test site should be appropriate for the intended application of the test material and should provide facilities for both:

a) open-air exposure, i.e. direct exposure to all atmospheric conditions and atmospheric contaminants;

b) sheltered exposure, i.e. exposure with protection from rainfall and solar radiation, either under a cover or in a partly closed space, such as shutter sheds, where the test specimens are also protected by shuttered side walls.

7.2 Comprehensive details of the shelter and the manner of exposing specimens shall be given.

NOTE Shelters eliminate washing of corrosion products by rainfall and can also impact on the surface temperature by limiting the rise of surface temperature associated with direct sunlight or by constraining a decrease for external cooler/windy conditions.

7.3 The results obtained under different designs of shelters shall be considered distinct.

7.4 Natural exposure testing of stress corrosion specimens should be based on the principles set out for corrosion testing in ISO 8565, with the test specimens mounted on frames of metallic or wooden construction in such a way that the rack material shall exert no influence on the corrosion of the test specimen.

7.5 The method of attaching specimens to the test frame shall prevent neighbouring specimens from touching, sheltering or influencing one another. There should be complete electrical insulation between the specimens and the test frame. If this is not possible, there shall be no electrolyte pathway from the specimen gauge area and metallic test frame components. Fixing elements may be made from inert and durable materials. Alternatively, bolts or screws, fitted with electrically insulating sleeves and washers, may be used. The area of contact between the test specimens and their holders shall be as small as possible and should be protected from the test environment.

7.6 The test frames shall be designed to control specimen orientation relative to the pollutant and contaminant source.

The specific orientation of the specimen depends on specimen type but should be optimised to maximise uniformity of deposit distribution and wetness on the most stressed region of the specimen.

7.7 The design of the frame shall be such that test specimens are not affected by water which runs off the test frame or other specimens, or by splash water from the ground. The minimum height shall be chosen to prevent both splashing by rainwater and burial in snow-drifts and should be not less than 0,5 m from ground level.

7.8 The following environmental data for the characterization of the atmosphere shall be obtained as specified in ISO 8565:2011, Annex A; specifically:

— air temperature, in degrees Celsius;

— relative atmospheric humidity (RH), as a percentage;

— amount of precipitation, in millimetres per day;

— sulfur dioxide deposition rate in accordance with ISO 9225, in milligrams per square metre per day, or concentration in micrograms per cubic metre;

— chloride deposition rate in accordance with ISO 9225, in milligrams per square metre per day.

7.9 Additional environmental data can be obtained (see ISO 8565:2011, Annex A for full details) including, but not exclusively, solar radiation, NO_2 concentration, O_3 concentration, HNO_3 concentration, particulate deposition, and measured on the same timeframe as specified in 7.8.

7.10 ISO 22858 should be consulted for guidance on the use of electrochemical methods of monitoring corrosion. ISO 9223 gives guidance on corrosivity. Both standards provide an indication of the relative aggressivity of the environment, and by implication the potential impact on stress corrosion cracking.

7.11 The overall test period should be long enough to account for seasonable variability to ensure the data are statistically meaningful. If specimen failure does not occur sooner, a minimum period of two years is recommended.

7.12 Sufficient specimens should be exposed to permit meaningful statistical variation from specimen to specimen to be accounted for, typically in triplicate. Where appropriate, the number of specimens exposed should allow specimens to be removed at intermediate times to elucidate information about the time evolution of damage, including time-averaged crack growth rate.

8 Testing in artificial environments

8.1 Laboratory testing shall be conducted in a climatic and/or corrosion chamber or in a sealed vessel, with the RH in the latter controlled by inclusion of an appropriate salt solution at saturation for the test temperature. The salt used in the latter shall be reported. The values of RH and temperature in the controlled environments should be constantly monitored and logged via a calibrated hygrometer.

8.2 ISO 22858 should be consulted with respect to the application of electrochemical methods for monitoring corrosion rate in the test chamber or vessel, should such in-situ measurement be desired.

8.3 The test temperature shall be maintained to ±1 °C and RH to within ±2 % when held at set values.

8.4 Deposition of test salt

8.4.1 The test may be conducted with the test salt deposited onto the self-loaded test specimen prior to exposure by a controlled droplet process or in a salt spray environment, as specified in ISO 9227.

8.4.2 The cleanness of specimen surface should ensure homogeneous surface wettability prior to salt deposition (see 5.6).

8.4.3 For the controlled droplet method, single or multiple droplets can be deposited using either a handheld pipette or automated dispensing system such as inkjet printing and multiprobe liquid handling system, with much greater control over quantity and distribution. Multiple droplets should also be evenly spaced to avoid overlap. Deposition by salt spraying prior to testing can also be adopted but there is less control of deposition density and salt layer thickness.

Testing may include the use of a second material to form a crevice or galvanic couple with the specimen.[1] Application of the salt solution should wet and fill the crevice.

8.4.4 The droplet should be formed by evaporating a droplet of salt solution delivered to the surface by a pipette or similar dispenser with the objective of achieving a relatively uniform distribution of deposit.

8.4.5 Methanol can be used as a salt carrier by virtue of the more rapid evaporation relative to aqueous solution. This evaporation can be accelerated by prior heating of the specimen to allow flash drying and, arguably, a more consistent salt distribution. Testing to establish the most effective method for optimising the uniformity of the salt distribution should be performed. SEM-EDS can be used to give an indication of uniformity of deposit.

8.4.6 Stress corrosion cracking testing may be conducted in a salt spray environment as a means for depositing salt on the test specimens and such testing should follow the methodology specified in ISO 9227.

NOTE While aggressive in terms of salt deposition, this test condition with constant RH can be less aggressive than a cyclic humidity test (see 8.6.8) with associated wetting and drying.[5] ISO 9227 is not commonly used for atmospheric stress corrosion cracking testing.

8.5 Salt chemistry

8.5.1 The salt chemistry should reflect the chemistry formed on the intended application or selected to provide a more aggressive form of that to ensure conservatism in testing, or to accelerate testing. When undertaking testing according to ISO 9227, the salt solution composition adopted should be as specified in that standard.

NOTE Chloride salt deposits are most commonly used as these usually provide the most aggressive agents for inducing stress corrosion cracking.

8.5.2 Artificial seawater (ASW), such as specified by ASTM D1141, can be used as representative of marine aerosols.

8.5.3 Test environments reflective of marine application are often based on $MgCl_2$, occasionally $CaCl_2$, solution as a substitute for marine salts as these readily form a concentrated salt solution at relatively low RH and as such are considered to represent a severe test.

NOTE The solution chemistry of seawater at low RH values close to the deliquescence point of $MgCl_2$ is often inferred to be close to concentrated $MgCl_2$. Sea-salt evaporites end up essentially as carnallite, $KMg_2Cl_3{\cdot}6H_2O$, acting chemically as $MgCl_2$, with NaCl deposited as salt crystals.

8.5.4 Tests can also be conducted with the pH of the initial solution lowered, or with ferric chloride added to the droplet solution for applications related to stainless steels.

NOTE These aggressive oxidising solution modifications stimulate initial corrosion activity. However, the effect is transient due to rapid reduction of hydrogen ions, or ferric ions, as the case can be. The solution chemistry is expected to change relatively quickly in response to the electrochemical reduction processes on the surface and to evolve to the natural chemistry associated with the corrosion process.

8.6 Setting the chloride deposition density

8.6.1 The chloride deposition density (CDD) adopted should reflect the range of possible chloride salt deposition densities on the exposed surface in service or used to assess the sensitivity of the metal or alloy to chloride content for the selected salt mix.

Formula (1) provides a basis for estimating the CDD (e.g. μg/cm²), expressed here as on plain stress corrosion cracking test specimens.

$$\rho_{\mathrm{Cl}} = \frac{V \times \left[\mathrm{Cl}^{-}\right] \times M_{\mathrm{Cl}}}{A} \tag{1}$$

where

ρ_{Cl} is the CDD expressed as μg/cm² for consistency with terms below;

V is the droplet volume, expressed in μl;

Cl^{-} is the chloride ion concentration of the droplet solution, expressed in mol/l;

M is the molecular mass of the chloride ion, expressed in g/mol;

A is the deposition area (expressed in cm²).

8.6.3 The area of salt solution should be of a size sufficient to provide sufficient cathodic current to support localised corrosion and stress corrosion cracking but should not be so large that it extends to edges of specimens or is affected by specimen curvature.

8.6.4 The target dimension of the deposition area should be defined, and the corresponding volume of solution determined as follows.

8.6.4.1 Using the same material with the surface identical in preparation to that for the stress corrosion cracking test specimen, the contact area of the salt film formed shall be measured very soon after the deposition, under a microscope for different volumes of the droplets. A series of measurements should be carried out to assess the repeatability of the process.

8.6.4.2 For the selected values of CDD, A and V, the required concentration of chloride in the droplet solution should then be estimated based on Formula (1).

8.6.4.3 After applying the droplet with the calculated salt concentration solution, the actual area of the deposited salt droplet shall be measured and the actual CDD recalculated.

The CDD value should only be targeted as an initial value. The migration of deposited ions as a result of corrosion and wetting and drying will result in local variation of the chloride concentration.

8.6.4.4 To increase CDD further a repeat application of the droplet to the existing salt area can be made.

NOTE 1 For the same droplet size, but different concentration of the same salt, the solution chemistry of the salt film is determined by the RH (> deliquescence RH). The thickness of the salt film is determined by the CDD, which increases with increased salt concentration of droplet solution. For a salt film solution in equilibrium at a given RH at or above deliquescence, the higher the RH the greater the solution volume and the lower the solution concentration.

NOTE 2 At the deliquescent RH for a mixed salt composition such as in ASW, a saturated solution forms but with a composition that reflects more the salt with the lowest deliquescent RH. Residual salt crystals of the species with higher deliquescence RH remain on the surface and can initiate corrosion via a micro-crevice mechanism. With increasing RH, the droplet size tends to increase with little change in contact area, the solution progressively dilutes, and the salt solution composition becomes more reflective of the mixed salt.

8.6.5 For testing with notched test specimens, and notched and precracked specimens, droplets should be deposited along the notch and the total volume recorded. Here, it is less readily possible to define CDD with confidence as measurement of the wetted salt deposit is less straightforward.

8.6.6 For specimens with crevices, the solution may be applied to the surface before placing the crevice forming material or applied to the edges of the crevice. For a crevice, it is less readily possible to define CDD and the applied solution volume shall be recorded.

8.6.7 The self-loaded test specimens with their deposited salts shall then be located in the testing chamber or vessel and the RH and temperature adjusted to the desired values.

8.6.8 Cyclic variation of the temperature and RH to enable exposure of the stress corrosion cracking test to wet and dry periods may be conducted as this provides a more severe testing condition. The proposed testing regime adopted should take account of the service application.

8.6.9 Sufficient specimens should be exposed to account for statistical variation from specimen to specimen and, where appropriate, to allow specimens to be removed at intermediate times to elucidate information about the time evolution of damage, including time to first observation of a crack and time-averaged crack growth rate if not using in-situ crack growth rate measurement methods (see 8.7).

8.7 In situ crack monitoring

8.7.1 In situ crack growth rate monitoring for surface-developing cracks can be carried out by the techniques highlighted in ISO 21153. Optical methods, including digital image correlation tend to be constrained by obscuration of corrosion product. The direct current potential drop (DCPD) method can be a more effective method but has challenges. Compliance methods that combine in situ measures of deflection with finite element modelling of the specimen can be used, but crack length estimates shall be validated, and estimates are dependent on crack path assumptions.

8.7.2 Minimising probe spacing is critical to optimising DCPD resolution of crack size measurement, as resolution falls off very rapidly with increasing probe spacing. A target spacing of not greater than 1 mm is recommended for small crack growth rate measurement, with the probes symmetrically positioned about the crack precursor and aligned along the stress axis. By implication, the probes would be within the droplet area and protection would be required. Some perturbation of the droplet by the probes is inevitable.

8.7.3 Pre-pitting, with or without fatigue precracking, can be performed to provide the crack precursor but the extent to which stress corrosion cracking develops can depend critically on exposure condition. Crack precursor development can be achieved by applied galvanic currents or by galvanic coupling. In testing simulating coupling conditions, the coupling process may be sufficient to stimulate stress corrosion crack initiation.

8.7.4 For notched fracture mechanics specimens, with or without fatigue precrack, with the salt deposit contained in the notch, the DCPD probes are simply attached on the surface on either side of the notch. The use of notched specimen can be an approach to crack growth rate measurement when the methodology for surface developing cracks proves inadequate. Non-uniformity of crack growth can occur in the early stages and the fracture surface on test termination should be inspected to assess continuity and uniformity of the crack front.

8.7.5 Monitoring of crack development in fracture mechanics specimens can be undertaken by other techniques such as clip gauges, back-face strain gauges, induction displacement sensors, and load cells as appropriate for the application.

9 Post-test analysis

9.1 Photographic images of the test specimen upon removal shall be taken with attention to all exposure surfaces.

9.2 Where appropriate to the specimen loading conditions, the final stress should be measured. The extent of stress relaxation during the course of exposure should be calculated.

9.3 Prior to any cleaning procedure of natural exposure specimens, the chemistry and thickness of surface deposits post-test should be determined, using scanning electron microscopy imaging and energy-dispersive spectroscopy for example. Since the chemical composition and deposit thickness may not be consistent over the whole period of testing, these variables should be characterised upon removal of specimens at any intermediate time.

9.4 To enable inspection for pits and cracks, corrosion products should be removed using a method appropriate to the alloy, as described in ISO 8407, and photographic images taken.

9.5 The incidence of cracking shall be measured by optical microscopy and metallographical examination. The data shall include numbers of specimens examined at particular exposure time with the same loading conditions, observation of pitting, maximum crack length observed in the group of specimens, number of cracks, and number of actual failures relative to the number of specimens examined. Measurement of maximum crack depth by serial layer removal or by 3D imaging techniques such as X-ray computed tomography (XCT) can also be undertaken.