



BSI Standards Publication

Corrosion of metals and alloys — Stress corrosion cracking

Part 11: Guidelines for testing the
resistance of metals and alloys to hydrogen
embrittlement and hydrogen-assisted
cracking

National foreword

This British Standard is the UK implementation of EN ISO 7539-11:2014. It is identical to ISO 7539-11:2013. It supersedes BS ISO 7539-11:2013 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee ISE/NFE/8, Corrosion of metals and alloys.

A list of organizations represented on this committee can be obtained on request to its secretary.

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English Version

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Foreword

The text of ISO 7539-11:2013 has been prepared by Technical Committee ISO/TC 156 “Corrosion of metals and alloys” of the International Organization for Standardization (ISO) and has been taken over as EN ISO 7539-11:2014 by Technical Committee CEN/TC 262 “Metallic and other inorganic coatings” the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2015, and conflicting national standards shall be withdrawn at the latest by June 2015.

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Endorsement notice

The text of ISO 7539-11:2013 has been approved by CEN as EN ISO 7539-11:2014 without any modification.

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Corrosion of metals and alloys — Stress corrosion cracking —

Part 11:

Guidelines for testing the resistance of metals and alloys to hydrogen embrittlement and hydrogen-assisted cracking

1 Scope

This part of ISO 7539 gives guidance on the key features that should be accounted for in designing and conducting tests to evaluate the resistance of a metal or its alloy to hydrogen embrittlement and hydrogen-assisted cracking.

NOTE Particular methods of testing are not treated in detail in this document. These are described in other International Standards to which reference is given.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 7539-7, *Corrosion of metals and alloys — Stress corrosion testing — Part 7: Method for slow strain rate testing*

ISO 17081, *Method of measurement of hydrogen permeation and determination of hydrogen uptake and transport in metals by an electrochemical technique*

3 Factors to be considered in hydrogen embrittlement and hydrogen-assisted cracking testing

3.1 Dynamic plastic straining

3.1.1 Surface films such as passive oxide films, and sulphide films in the case of exposure of carbon steel to H₂S environments, for example, can markedly reduce hydrogen uptake. Film rupture will enhance ingress locally, which means that dynamic plastic straining and the strain rate can be particularly important. In that context, there is then usually no relationship between hydrogen uptake as measured in a permeation experiment and the cracking response since uptake is local at the film rupture sites. A possible exception is when there is a significant sub-surface region of susceptibility associated with residual stress or microchemistry as might possibly be found in welds. Here, detailed characterization of the weld should be conducted prior to testing.

3.1.2 Dynamic plastic straining may be induced under static load if there is significant creep, as in some duplex stainless steels.

3.1.3 In testing of alloys that are actively corroding, there is often a correlation between cracking and the measured bulk hydrogen uptake. Dynamic plastic straining may have only a relatively minor role in hydrogen uptake in that case.

3.1.4 In all alloys, dynamic plastic straining and the strain rate may be important in dislocation transport of hydrogen. The mobility of hydrogen atoms and trapping at dislocations means that dislocations can

move hydrogen (though constrained by microstructural boundaries) and possibly deposit the hydrogen at susceptible sites, e.g. grain boundaries.

3.2 Test time and hydrogen uptake

3.2.1 Hydrogen atoms are mobile and can diffuse to sites of potential susceptibility, which may be some distance from the primary source. A fundamental question is how long should a laboratory test be to ensure that hydrogen uptake is sufficient in reflecting behaviour in service, for which exposure times are of the order of years. The critical issue is the location of cracking with respect to the primary source of hydrogen. If the latter is remote, then test times need to reflect this. Hence, hydrogen diffusivity and test time are important. In delayed hydrogen cracking, for example, analysis of the hydrogen distribution with time in response to concentration and stress gradients may be necessary to assess the likelihood of cracking in service.

3.2.2 The location of cracking will be system-dependent. It may be associated with mid-thickness of a low-alloy carbon steel pipeline with centre-line segregation. If using a pre-cracked specimen, it is self-evidently local to the crack tip. In a weld, it could be sub-surface.

3.2.3 The primary source of hydrogen is most likely at a locally strained region if testing corrosion-resistant alloys in the passive state because film rupture sites provide the main points of entry. In this case, test times may be relatively modest unless testing under conditions of pitting corrosion (or crevice corrosion). The local aggressive chemistry associated with pitting and crevice corrosion, together with the dissolution of protective films, will encourage hydrogen uptake. If the crack initiates from a pit, pit size may be a factor and, thus, there may be an effect of exposure time specific to that aspect. Failure may not be expected unless above the critical pitting/crevice temperature, though there could be an effect of stress on the value.

3.2.4 For systems with no protective film, the primary source of hydrogen is a complex function of the solution chemistry and applied potential. If there is a species in the bulk solution that enhances hydrogen generation and absorption but is depleted in a crack, then the primary source is the external surface exposed to the bulk solution. Examples are acid solutions and solutions containing hydrogen sulphide. However, in H₂S environments, the formation with exposure time of an iron sulphide film on the exposed external surface will progressively lead to a reduction in hydrogen entry and may change the locality of the primary source to that of the crack tip.

In less “aggressive” or gaseous environments, hydrogen uptake at the crack tip may be favoured. When using pre-cracked specimens with cathodic protection potentials at sacrificial anode values, the primary source of hydrogen is from the external surface because of potential drop and chemistry changes in the crack.

3.3 Temperature

3.3.1 Embrittlement is often associated with hydrogen trapping. Increasing the temperature tends to decrease trap occupancy but this may be compensated by increased kinetics of hydrogen generation and solubility in most materials. Diffusivity will also increase with temperature, and when comparing test results at different temperature, misconceptions about susceptibility can arise if the hydrogen uptake is not at steady-state and the different levels of hydrogen ingress are not accounted for. For unprotected corrosion resistant alloys in the passive state cracking may occur only above a critical temperature associated with localized attack as noted in [3.2.3](#). Also, since the inherent ductility of metals tends to increase with increasing temperature, temperature will be expected to have a complex effect on embrittlement.

3.3.2 Testing should reflect the range of temperatures expected in service. It is important to recognize that for cathodically protected alloys, the most severe temperature may be the lowest temperature because this encourages trapping (see Introduction).