
**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**

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

*Partie 11: Lignes directrices pour les essais de résistance des métaux
et alliages à la fragilisation par l'hydrogène et la fissuration assistée
sous hydrogène*



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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2. www.iso.org/directives

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The committee responsible for this document is 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*:

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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

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

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 7539-11:2013

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).

3.3.3 The extent of information on the effect of temperature transients is limited. These can be important if the cooling rate is relatively rapid compared with the rate of hydrogen egress from the metal. For many alloys, the lattice hydrogen solubility increases with temperature and the trap occupancy decreases. The ductility also increases. Thus, at sufficiently elevated temperature, there may be significant hydrogen uptake but no cracking. However, problems can arise if the rate of cooling is fast relative to diffusion. In certain steels, hydrogen may precipitate out of the lattice at interfaces as molecular hydrogen and raise the prospect of pressure-induced cracking. More generally, hydrogen atoms in the lattice will drop into trap sites. Combined with reduced ductility, cracking may ensue.

4 Selection of test method

4.1 General

A wide range of test methods have been developed that can be used to assess the resistance of materials to hydrogen embrittlement and hydrogen-assisted cracking. The Foreword lists a number of International Standards that are applicable to environment-assisted cracking in general, including both stress corrosion and corrosion fatigue. The electrochemical method for hydrogen permeation (ISO 17081) gives guidance in measuring hydrogen uptake and diffusivity. Additional test methods related to hydrogen embrittlement and hydrogen-assisted cracking, mostly for system-specific applications, are included in a complementary list in the Bibliography. In a number of applications, the loading and environmental exposure conditions are sufficiently straightforward and the purpose of the International Standard so constrained that additional guidelines in testing are not critical. However, for non-prescriptive test methods, the issues raised in [Clause 3](#) need to be accounted for in defining the test.

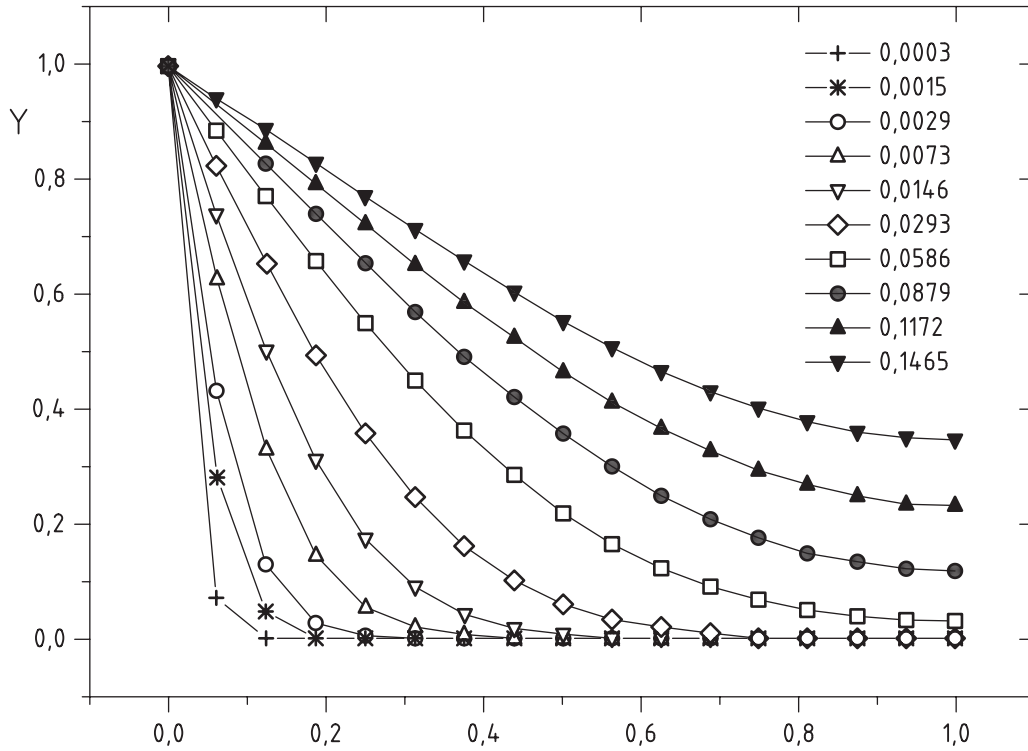
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4.2 Specimen type

The adoption of the specimen type in this respect depends on the design and maintenance philosophy in relation to the expectation of significant surface defects and their evolution with time. In addition to high local stresses in excess of yield, notched or pre-cracked specimens have the additional feature that the hydrostatic stress component localizes the hydrogen. Hence, the concentration of hydrogen is increased locally. However, the notch is wholly arbitrary in terms of depth, root radius, and, in the case of welds, the location of the root with respect to the heat affected zone (HAZ). There has been insufficient study of such testing and no guidelines are available. There is no agreed International Standard for hydrogen embrittlement testing in relation to the notch details and no guidelines are available. A notch will obviously enhance the likelihood of failure. Pre-cracked specimens can be used for ranking but are more commonly used to derive threshold stress intensity factors and crack growth data.

4.3 Test duration

4.3.1 The test duration should be based on the principles and considerations in [3.2](#) but pre-charging may be pertinent to ensure that uptake of hydrogen is significant. For some circumstances, such as cathodic polarization, it is relatively straightforward to obtain an approximate estimate of the time evolution of the hydrogen concentration, using Fick's second law with an effective diffusivity. An illustration of the predicted time evolution of the hydrogen concentration in a cylindrical specimen typical of a slow strain rate specimen during cathodic charging is shown in [Figure 1](#). Here, a is the radius, r is the distance from the surface, C_0^{av} is the surface concentration of hydrogen, and τ is a dimensionless time ($t \cdot D_{eff} / a^2$), where D_{eff} is the effective diffusion coefficient and t is time.



Key
 Y C/C_0^{av}
 X r/a

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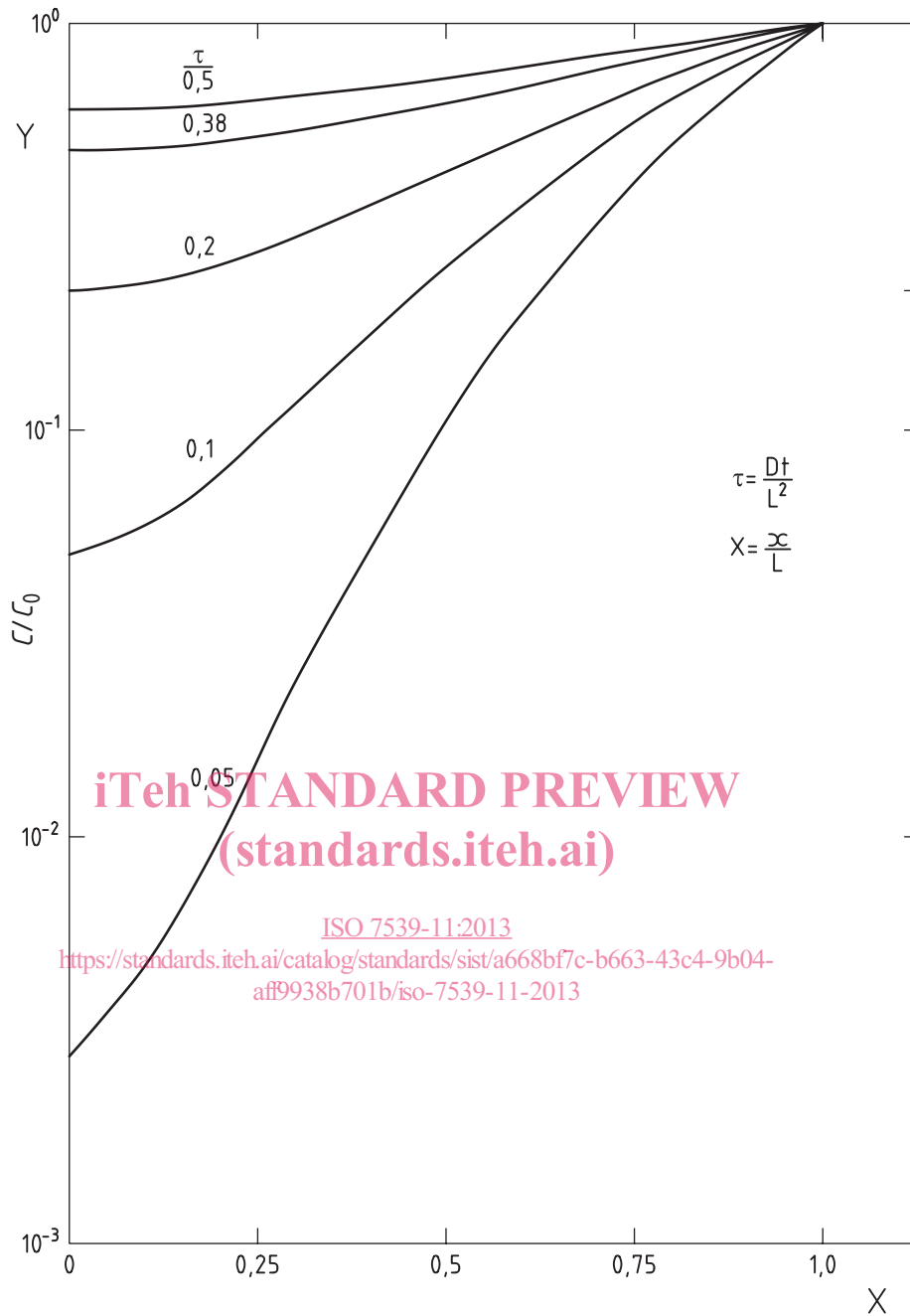
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Figure 1 — Normalized hydrogen profiles versus normalized depth in a solid cylindrical specimen based on solution of Fick's law

The profiles are shown for different values of the normalized time τ ($D_{eff} t/a^2$, where a is the radius of cylinder).

The test duration or pre-charging time may be such as to attain a steady-state hydrogen concentration, but since cracking initiates usually from the surface in these types of tests and the critical flaw size for unstable crack growth may be small, it may be considered more pragmatic to select a value close to that at some distance from the surface (e.g. 80 %) at r/a of 0,2. There is an element of judgement in the latter aspect that represents a balance between conservatism and pragmatic test times. The concentration profiles in plate specimens, based on solution of Fick's law, are shown in [Figure 2](#). These profiles can be used as a basis for assessing approximately the extent of through-thickness charging of a compact tension specimen, neglecting the presence of the crack. This can be used as a guide in pre-charging, for example.



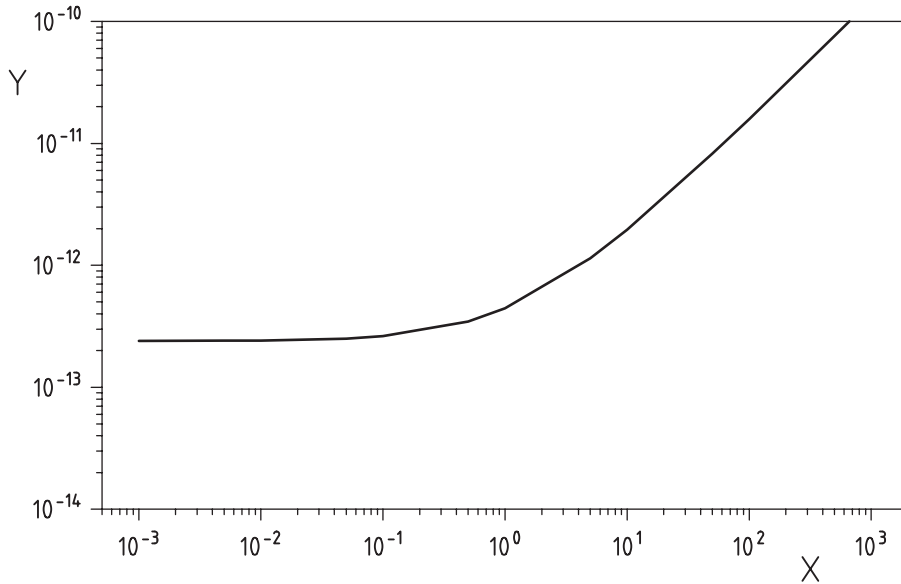
Key

- Y C/C_0
- X x
- D diffusion coefficient (effective diffusion coefficient in this case)
- L half thickness of specimen
- t time

NOTE The mid-thickness of the specimen is at position $X = 0$.

Figure 2 — Normalized hydrogen concentration profiles in a plate specimen that may be used to typify a compact tension specimen for which the primary source of hydrogen is from the external surface, at position $X = 1.0$

4.3.2 The effective diffusivity of hydrogen in metals is a function of the hydrogen uptake and, thus, exposure conditions, because of varying trap occupancy, as exemplified by Figure 3. Accordingly, measurements should be made under the exposure conditions and temperature of practical relevance and not abstracted arbitrarily from the literature. Guidance on measurement is given by ISO 17081.



Key
 Y $D_{\text{eff}}/\text{m}^2 \text{ s}^{-1}$
 X C_0/ppb

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Figure 3 — Plot of calculated values of D_{eff} as a function of the sub-surface lattice concentration of hydrogen in parts per billion by mass, C_0 , for super 13 Cr steel at 23 °C

4.3.3 Examples of diffusivity at ambient temperature for some low-alloy steels under cathodic protection conditions are given in Table 1 and give an indication of the range of values.

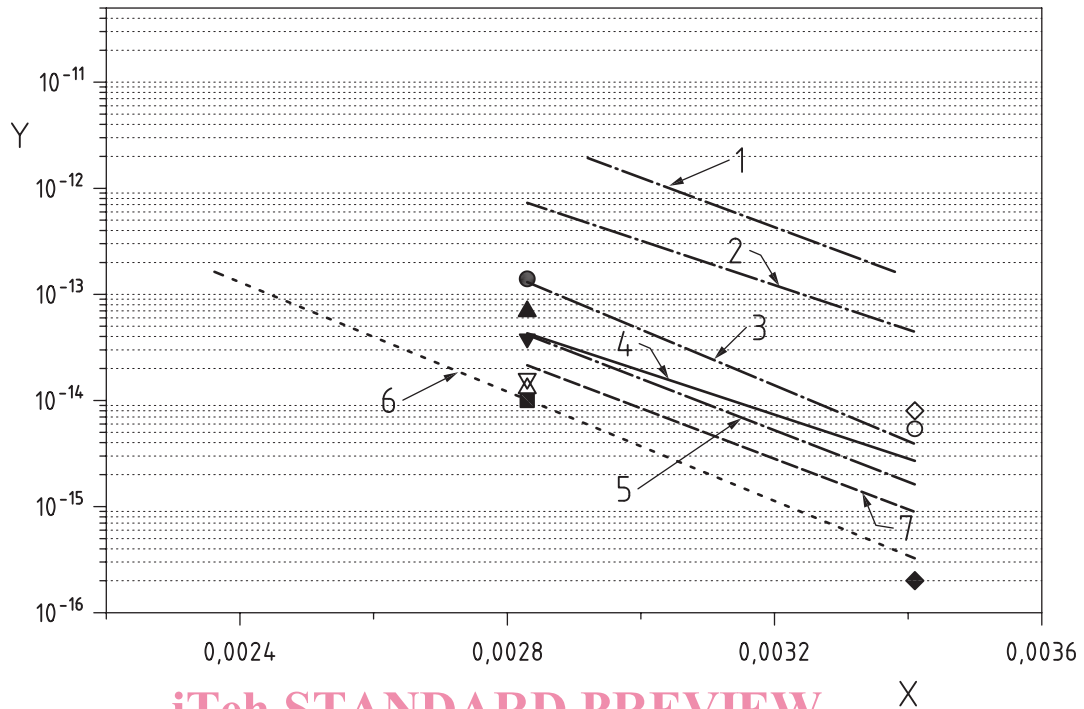
Table 1 — Hydrogen atom diffusivities for various low-alloy steels under cathodic protection at ambient temperature

Material	$D_{\text{eff}} (\text{m}^2 \text{ s}^{-1})$
Pure iron	$7,2 \times 10^{-9}$
BS 4360 50D (S355J2G3, 1.0577)	$1,7 \times 10^{-10}$
AISI 4340 (1.6565)	$1,7 \times 10^{-11}$
3,5 % Ni-Cr-Mo-V	$5,3 \times 10^{-12}$

For 25 mm thick pre-cracked specimens, the time to steady-state through-thickness hydrogen charging for the lower diffusivity alloy could be nearly a year for the 3,5 Ni-Cr-Mo-V steel. Pre-exposure is then pertinent. To minimize test time, it is useful to test with a relatively thin specimen within the constraints of achieving predominantly plain strain conditions. The benefits of that are apparent, for example, in the use of so called “half-thickness” double cantilever beam (DCB) specimens in testing carbon steels for sour oil and gas applications.

4.3.4 The literature should be consulted for diffusivity data for the alloy of interest. For certain corrosion-resistant alloys under cathodic protection, Figure 4 gives an indication of typical values. The activation energy is not intrinsic but will also depend on the exposure conditions so these data should only be used as a rough guide. While such data may not be so relevant if hydrogen is generated locally, there may be instances where bulk hydrogen uptake at high temperature is significant but the system is then cooled.

Also, it is always advisable to conduct a test with pre-charging to validate the perspective of predominantly local hydrogen uptake.



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Key

- Y $D_{eff}/m^2 s^{-1}$
- X $1/T$ in K^{-1}
- 1 super 13 Cr
- 2 Ni
- 3 alloy G
- 4 22 Cr
- 5 C - 276
- 6 AISI 300
- 7 25 Cr
- 245 SMO
- ◆ 82T SS
- alloy 600
- alloy 600
- ▲ alloy 750 SA
- △ alloy 750 DirA
- ◇ alloy 750 SA
- ▼ alloy 718 SA
- ▽ alloy 718 DirA

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Figure 4 — Temperature dependence of effective diffusivity for various corrosion-resistant alloys under cathodic protection, for illustrative purposes to indicate the range of values for the effective diffusivity and its dependence on alloy type [41]

4.3.5 In calculating hydrogen uptake and indeed in testing materials, non-uniform charging may need to be considered. For example, hydrogen-induced cracking of low-alloy and carbon steel pipelines in sour environments occurs internally, often in regions of centre-line segregation of MnS inclusions,