INTERNATIONAL STANDARD

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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

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

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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*:

- *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*

INTERNATIONAL STANDARD **ISO 7539-11:2013(E)**

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 H_2S 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 H_2S 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

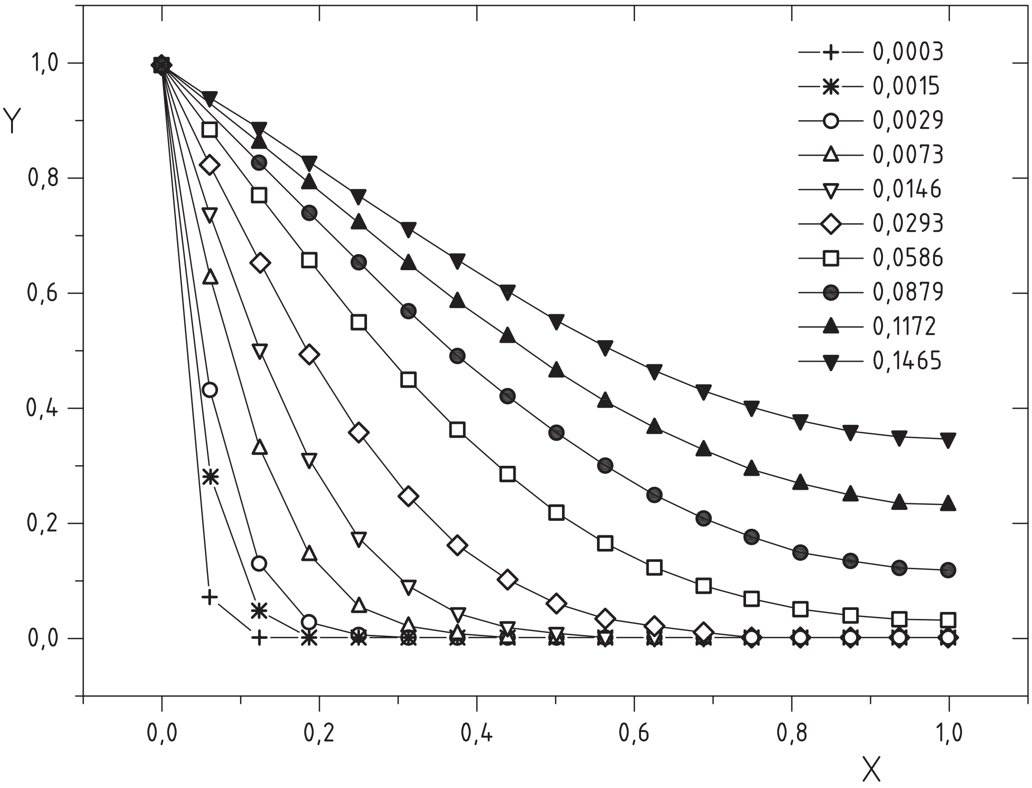
A wide range of test methods have been developed that can be used to assess the resistance of materials to hydrogen embrittlement and hydrogen-assisted cracking. The Foreword lists a number of International Standards that are applicable to environment-assisted cracking in general, including both stress 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.

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, $C_0{}^{\mathrm{av}}$ is the surface concentration of hydrogen, and τ is a dimensionless time ($t.D_{\mathrm{eff}}/a^2$), where D_{eff} is the effective diffusion coefficient and t is time.



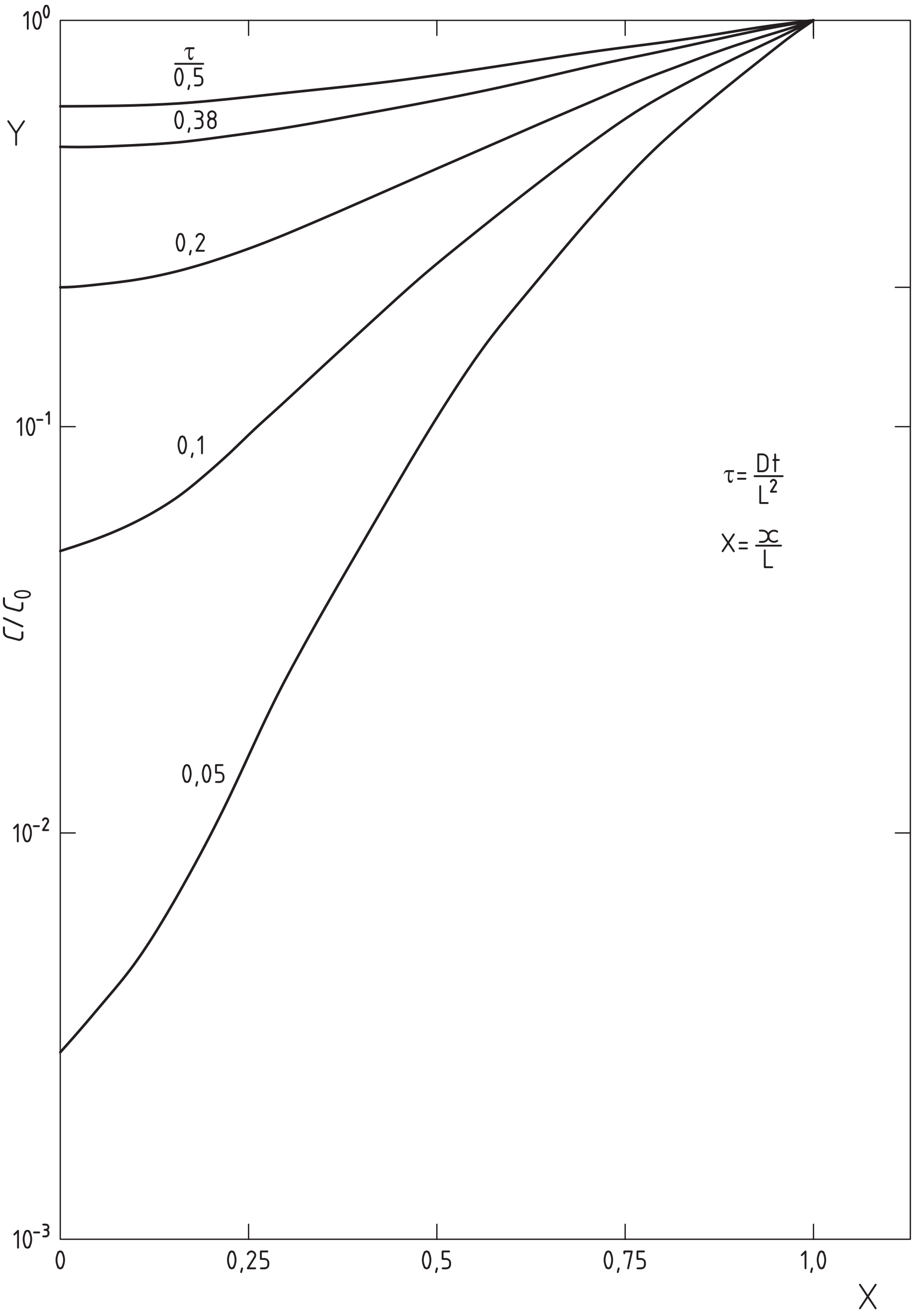
Key

Y	C/C_0av
X	r/a

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 ($D_{\text{eff}}\, t/a^2$, 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.



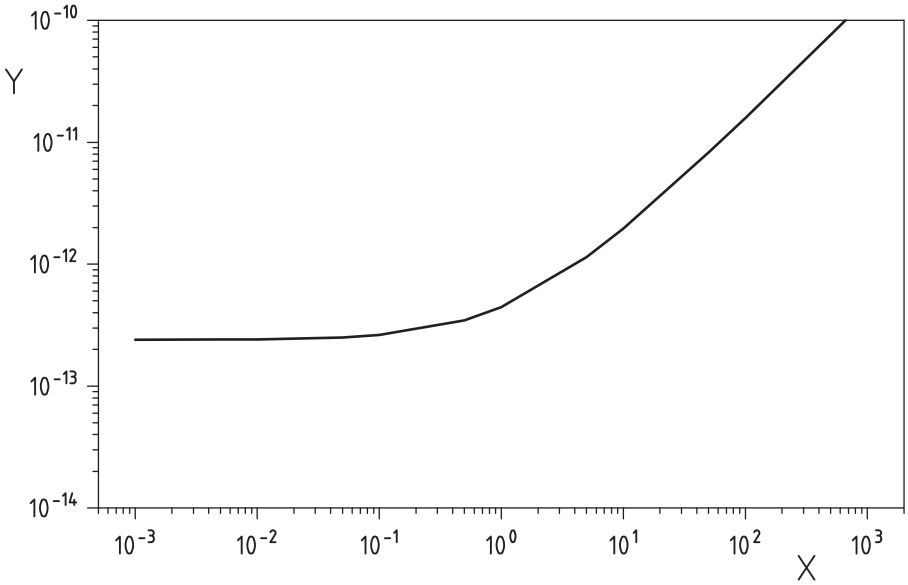
Key

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_{eff}/m² s⁻¹

X C_0/ppb

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.

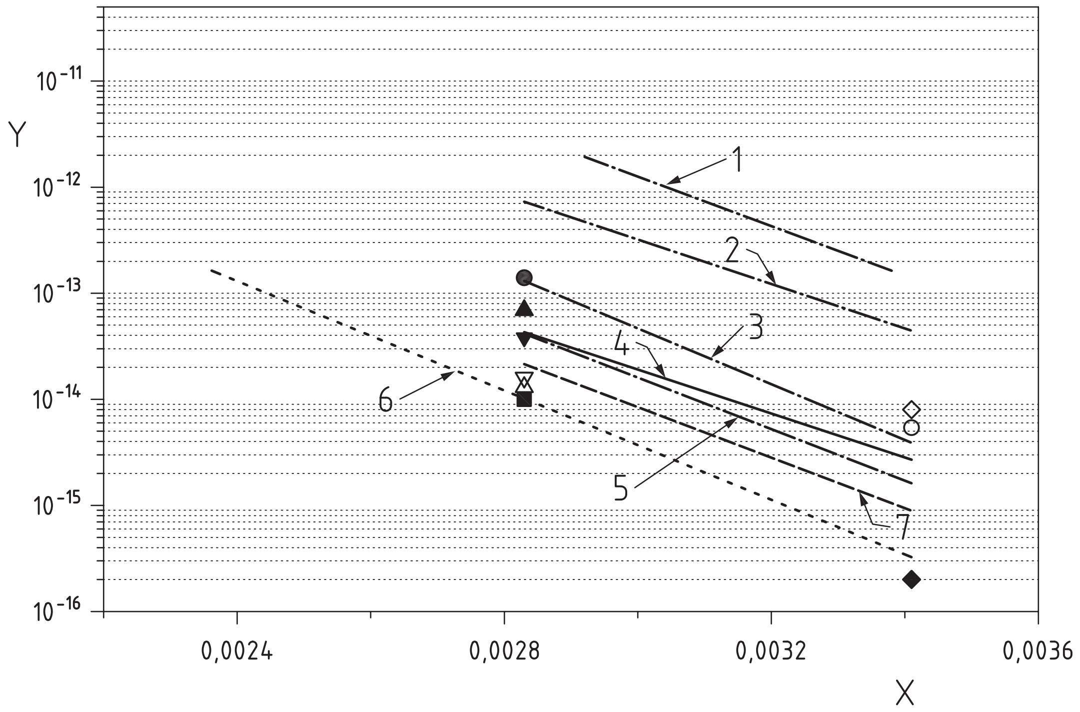
Table 1 — Hydrogen atom diffusivities for various low-alloy steels under cathodic protection at ambient temperature

Material	D_{eff} (m^2 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.



Key

Y	$D_{\text{eff}}/\text{m}^2\ \text{s}^{-1}$
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

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, though modern steelmaking practice is gradually eliminating this segregation. In testing of such pipeline material, allowance should be made for steady-state conditions of hydrogen charging to be achieved and the charging conditions at surfaces external and internal to the pipe should reflect service conditions. The latter is emphasized as the external surface of the pipe carrying sour crude or gas may be cathodically protected or may have corrosion product. Both can affect the hydrogen concentration gradient and may need to be simulated in testing.

4.3.6 In specimens that have been electroplated or welded and then exposed to a benign environment, there can be long-term migration of the hydrogen to sites of local high residual stress, for example. When this is coupled in the case of welds with exposure to an external source of hydrogen, determination of appropriate test times can be even more problematic. The best that can be done is to make calculations based on anticipated diffusivities and to examine the effect of test time (or pre-load hold times) on susceptibility.

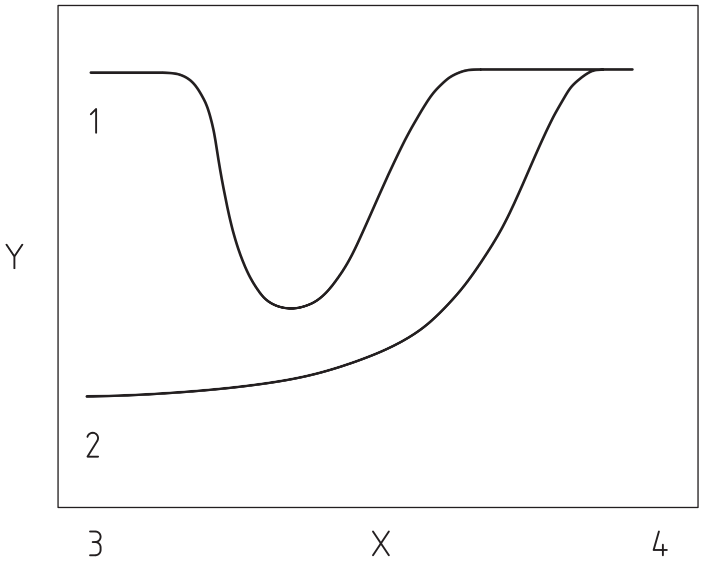
4.4 Load form

4.4.1 General

The loading should reflect the likely conditions in service but should allow for feasible upsets. Dynamic plastic strain is often applied in hydrogen embrittlement testing as a severe test for ranking purposes but it can be easily overlooked that such dynamic straining may arise in service, at least transiently, from a variety of sources including thermal transients in pressure vessels, soil or seabed movement in relation to pipes and so on. Cyclic loading is usually more apparent and is dealt with effectively in the International Standards on corrosion fatigue, ISO 11782-1 and ISO 11782-2. The issue in laboratory testing is how to accommodate the often poorly defined dynamic strains that may be experienced in service. In appropriate cases, attention may need to be given to multi-axial loading as the sensitivity to embrittlement can be different with different loading modes.

4.4.2 Plain specimens (including welds)

4.4.2.1 Testing should be conducted under conditions of rising displacement (or rising load in some cases) using ISO 7539-7 or NACE/TM 0198 as the reference standard. The strain rate is the key variable as shown in Figure 5, though the response can be system-sensitive.



Key

Y	reduction in area (%)
X	strain rate
1	Case 1
2	Case 2
3	low strain rate
4	high strain rate

Figure 5 — Schematic illustration of strain rate response of plain specimen with Case 2 being more closely associated with hydrogen charging conditions and Case 1 being more related to stress corrosion conditions

In the case of Figure 5, the response to strain rate will depend on the material and exposure conditions. Case 1 is more typical of stress corrosion cracking, but it would also represent a situation where hydrogen embrittlement is the failure mechanism, but corrosion plays a key role in generating the hydrogen. Case 2 may be more relevant to a cathodically protected system, though it is not clear whether some recovery might occur if the strain rate were made even lower.

4.4.2.2 At lower strain rates, the test time is inherently longer. This could be a factor when bulk hydrogen uptake is important and charging time is potentially a factor. It is then necessary to distinguish the effect of exposure time from that of the strain rate. Tests with pre-exposed specimens should be considered in distinguishing these effects.

4.4.2.3 In selecting the strain rate when testing of plain or notched specimens using ISO 7539-7, a value of 10^{-6} s^{-1} is often used in the first instance to assess relative susceptibility of materials or to compare relative aggressivity of environments. Although there are no generally agreed qualification criteria, if the strain to failure is close to yield, the material would be considered susceptible.

4.4.2.4 In many cases, the relative strain to failure may be quite large at this strain rate and beyond strains that may be experienced in service. Where there is an anticipated risk, it is prudent to test at lower strain rates. For corrosion-resistant alloys under cathodic protection, testing to 10^{-8} s^{-1} should be considered. To accelerate testing, a higher strain rate of 10^{-6} s^{-1} should be adopted in the elastic region and then switched to the slower strain rate close to yield. Typically, a 30-day test would result in more than 2 % plastic strain, which might be considered the upper limit of possible transient service strain. If there is no fracture, the specimens should be inspected metallographically for microcracks.

4.4.2.5 In assessing the significance of the slow strain rate data for service, the history of the system is important. If the material has been strained in service plastically at a displacement rate (and total strain value) at which cracks are not initiated, there can be work-hardening. Correspondingly, if the material is then subjected to a very slow displacement rate, the stress to cause plastic deformation will be greater. The material will be more resistant to crack initiation.

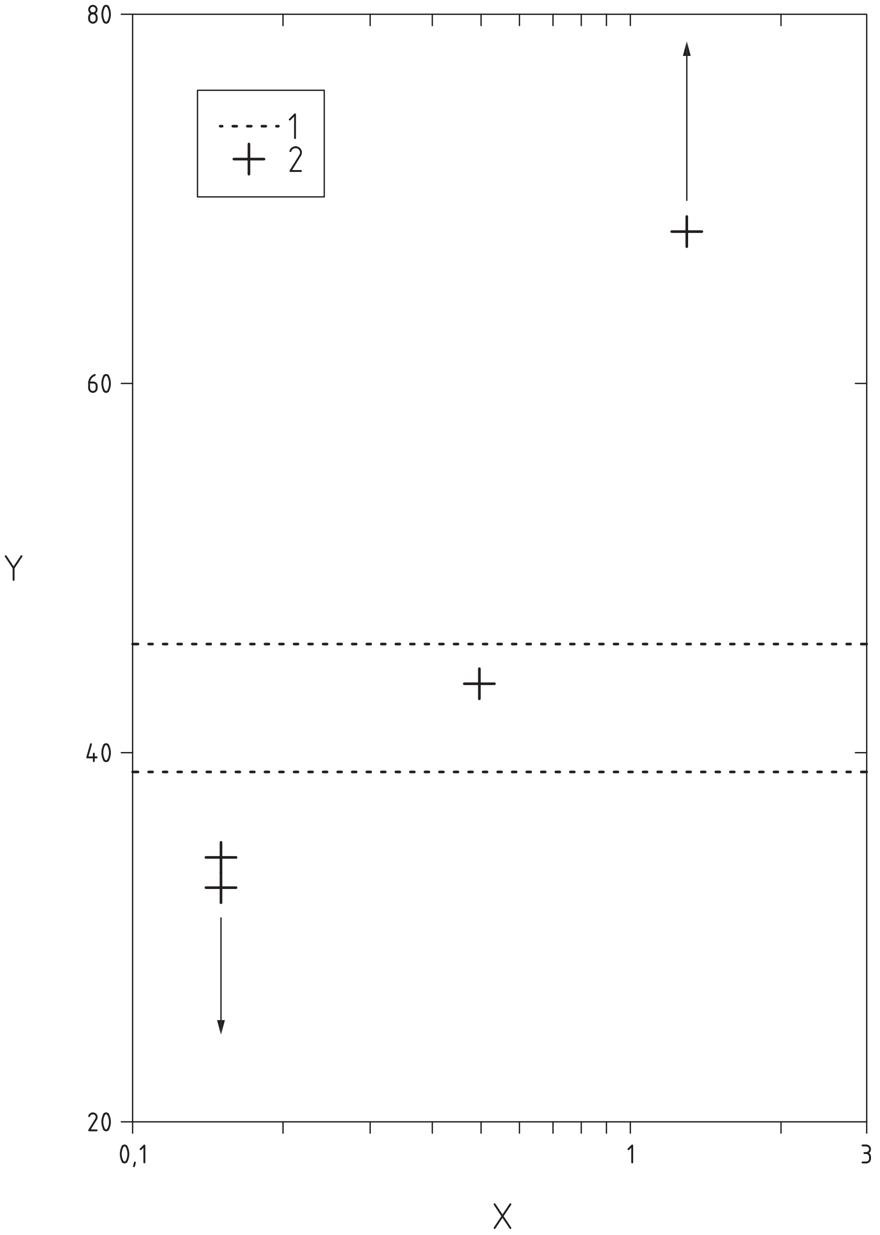
4.4.3 Fracture mechanics specimens

4.4.3.1 Most testing is conducted with Mode I loading, but it is important to recognize that embrittlement can be sensitive to the loading mode and significant components of shearing modes, II and III, may result in a different response. When testing with fracture mechanics specimens for the determination of K_{ISCC} or K_{IH} (Mode I), where K_{ISCC} is the threshold stress intensity factor for stress corrosion cracking and K_{IH} is the threshold stress intensity factor for hydrogen stress cracking, the value of the threshold will often decrease with decrease in the displacement rate across the crack mouth, though again it may level off to a strain rate insensitive value if the strain rate is low enough. This levelling off has been observed for an aluminium alloy but there are not enough measurements to generalize.

Figure 6 shows an example of the displacement rate sensitivity of an AISI 4340 steel corroding in seawater. For this system, the source of hydrogen is most likely to be at the crack tip so the lower threshold at the lowest strain rate is not explained by the increased test time. If the same steel were to be tested with cathodic protection, the primary source of hydrogen could be on the surface external to the crack and strain rate and exposure time could both be important. Pre-charging would be pertinent.

4.4.3.2 Testing of pre-cracked specimens under rising displacement or load is not uncommon but is more expensive because of the requirement for more complex specimens and instrumentation. It should be undertaken where a significant defect is expected in service and should be conducted over a range of crack mouth displacement rates to obtain a minimum K_{ISCC} (K_{IH}). The step-wise rising load test can be a useful preliminary test to give an approximate value of the threshold. This gives guidance on the initial K value at which to start the slow rising displacement or rising load test.

In these tests, judgement is required in defining acceptance criteria with respect to the value of K_{ISCC}. As with notches, the location of the pre-crack in a weld may be critical and may need to be systematically examined.



Key

Y K_{ISCC} (MPa m $^{1/2}$)

X displacement rate across crack mouth (μm/h)

……. constant load tests

\+ rising displacement tests

NOTE Testing based on ISO 7539-9.

Figure 6 — K_{ISCC} vs load-line displacement rate for an AISI 4340 steel actively corroding in ASTM seawater at 20 °C

4.4.3.3 It should be noted that determination of K_{ISCC} (K_{IH}) can be made with DCB or wedge open loaded (WOL) specimens for which K is decreasing. Here, there is an inherent dynamic plastic strain at the crack tip associated with the crack growth process itself. Of course, this is uncontrolled and does not reflect transient loading issues in service.

4.4.3.4 In cases where the applied stresses are high in relation to the material's yield strength and plastic deformation exceeds the assumptions of linear elastic fracture mechanics, the analysis based on the linear elastic fracture mechanics (LEFM) concept is inadequate. As an approximate approach, K_{QSCC} (provisional value for the threshold stress intensity factor when plain strain conditions are not fully met and becomes equal to K_{ISCC} when the validity criteria for plane strain conditions are fully satisfied) may be used (see ISO 7539-6 and ISO 7539-9). However, this requires that the testing conditions in terms of specimen thickness, for example, are in service. A more formal treatment involves adopting elastic-plastic fracture mechanics parameters such as the crack-tip-opening-displacement, CTOD (e.g. in the form of the directly measured δ_5, see BS 7448-1), the J-integral or the crack-tip-opening angle (ψ), with the appropriate threshold values for the CTOD, and/or the J-integral at the onset of crack growth. These parameters are normally used to characterize the fracture behaviour of ductile materials in air or inert environments and should be applied instead of the stress intensity factor, K_I. In this case, the time variation of the crack extension, Δa, is determined as a function of δ_5 or J. An advantage of adopting δ_5 is that it is equally suited to LEFM and can be directly measured on a laboratory specimen as well as the component to be assessed, where K solutions may not be available. Also, the thickness of the component does not play a role in this case.

4.5 Pre-charging and hydrogen effusivity

4.5.1 The generic aspects of environment control are covered in the various International Standards. A distinctive aspect in relation to hydrogen embrittlement is the extent to which pre-charging is undertaken and the conditions maintained during and after pre-charging. The duration of pre-charging should be assessed on the basis of the evolution of hydrogen in the alloy in relation to the location of cracking (see 4.3).

4.5.2 Pre-charging should only be done in the test conditions to which the specimen is then going to be exposed. It is carried out without load, often in a cell with other specimens. In most cases, this involves cathodic polarization but it could be achieved by exposure to hydrogen gas or under freely corroding conditions, for example. The key stage following any pre-charging is removal of the specimen and loading in the test environment. Loss of hydrogen by diffusion can be rapid in some low-alloy carbon steels. Provided the test is carried out in the same test environment, this loss will be recovered quickly and should not be an issue.

4.5.3 However, if testing in air, perhaps after thermal charging, the loss of hydrogen may be significant. This is true for all alloy systems when using fracture mechanics specimens. Hydrogen effusion at the crack tip, since hydrogen concentration gradients are steep, may occur because the important process zone size has dimensions of only a few microns. The crack tip strain disrupts the surface film (including cadmium plated films) allowing hydrogen to exit and recombine more easily. The loss of hydrogen will be continuous through the test. Misleading results may be obtained.

4.5.4 Pre-exposure at elevated temperature, corresponding to maximum value in service and mechanical testing at lower temperature (with sufficiently rapid cooling), would be pertinent for some applications where retained hydrogen is unable to rapidly effuse from the material.

4.6 Testing of welds

4.6.1 The testing of welds for resistance to hydrogen embrittlement offers specific challenges in relation to hydrogen uptake, the type of specimen, and residual stress.

4.6.2 If there is a sub-surface defect such as a microcrack, then it may take some time for hydrogen generated at the surface to transport to the site. For some corrosion-resistant alloys with very low hydrogen atom diffusivity, such as duplex stainless steels, this could mean years. Test times are also difficult to predict because the hydrogen diffusivity and solubility in the weld metal, heat affected zone, and parent material will vary. Such very long testing is not realistic and can only be circumvented by identification of the defect and its position so that specimens could be manufactured with the defect close to the surface. This is not easy as it would mean non-destructive evaluation of every weld prior to testing, an unlikely scenario. Thermal pre-charging is not useful because the hydrogen uptake will not be representative of service exposure. Thus, there may be limitations in characterizing the performance of some welded corrosion-resistant alloys.

4.6.3 Specimens

4.6.3.1 In selecting the form of the test specimen, the issues are the use of plain specimens (i.e. fully machined) versus specimens with the relevant surface in the as-welded state or the adoption of notched or pre-cracked specimens. There is currently no consensus.

4.6.3.2 Plain, fully machined, welded specimens can be used for relative comparison of performance where the key factor is repeatability. Such testing is less satisfactory as a qualification test. It is useful to conduct a detailed hardness and microstructure characterization prior to testing in order to assess the extent of variation, give guidance on specimen preparation, and identify any possible influence on test results. There may also be variations in residual stress through the thickness. Accordingly, specimens should be removed from the source material, e.g. pipe, in a consistent way. The surface should be prepared to a repeatable finish as agreed with interested parties but usually with an R_a value ≤ 0,25 µm. The test specimen shall be fabricated carefully at an appropriate rate to avoid overheating and unnecessary cold working of the surface. If a lubricant is used, this could affect the surface chemistry of the specimen. The lubricant should be cleaned from the surface of the specimen using a suitable solvent and the specimen dried in cold air.

4.6.3.3 When testing with one surface in the as-welded state, machining from one side only may often result in a variation in thickness on either side of the weld and the extent of this should be recorded. This variation in thickness will cause non-uniform straining of the specimen. The virtue of such testing is that it retains the characteristics of the surface in terms of the nature of the oxide and stress concentration factors associated with the transition from weld to parent plate or with small defects. The surface should be cleaned with a suitable solvent but otherwise reflect service condition. Such testing is usually based on four-point bend, C-rings, or flat tensile specimens. The "flat" tensile specimen is particularly useful for slow strain rate testing but it is important in presenting the results to allow for the varying thickness along the specimens when defining stress and strain.

4.6.3.4 The use of notches in otherwise plain welded specimens has its proponents because it generates a controlled defect. However, there is no standardization of the notch depth or root radius. Further, the location of the notch tip has to be considered. This should be located in the region of greatest sensitivity in terms of the microstructure; otherwise, a misleading result may ensue. This may only be determined by systematic testing with the notch tip in varying locations.

4.6.3.5 The adoption of a fracture mechanics approach (see 4.4.3) also has support for the same reason as for notches but with the advantage of a clear analytical framework. The problem then is as with the welds, where to locate the crack. There is also the issue of the depth of crack in relation to any variation in hardness and residual stress through the material, a factor often overlooked.

5 Post-test evaluation

The common procedures for post-test evaluation for different types of test are detailed in the relevant standards. The distinctive feature of hydrogen is the retention in the alloy at the end of the test after removal from the test environment. If the specimen is still under load (e.g. DCB specimen), any crack may continue to grow.

In NACE/TM 0177-96, the DCB test is conducted for a specified time. The specimen is then removed from the solution but the specimen may not be unloaded for some time. There may then be some further crack extension due to the retained hydrogen and this needs to be differentiated from the crack extension in solution. On pulling the specimen to fracture, the loading rate should be high to ensure no hydrogen-assisted crack growth.

In other tests, the DCB test may be conducted until the growth rate falls below some value that may be considered a threshold. Experience has shown that after testing at elevated temperature to the “threshold” value, a crack may grow at an accelerated rate for a period after the temperature is returned to ambient conditions. Hence, the specimen should be removed from the solution as quickly as possible at the relevant temperature and rinsed to mark the position of the crack front at temperature. The load should be removed as soon as possible and specimen then pulled to fracture at a high rate.

Similarly, in pulling to failure tensile or bend specimens after exposure, the strain rate should be of the order of 10^{-3} s^{-1} to ensure that no further hydrogen-assisted crack growth associated with retained hydrogen is artificially induced.

It is useful also to examine the fractographic appearance of any failed specimen as this may give an indication of sub-surface defects, in the case of welds, and would provide a reference for comparison with any failures experienced in service.

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