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Standard Test Method

Four-Point Bend Testing of Materials for Oil and Gas Applications

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Foreword

Four-point bend testing is used extensively in the oil and gas industry to evaluate resistance of metals to sulfide stress cracking and stress corrosion cracking. The surface of the specimen to be exposed to the environment in service is stressed in tension and the other surface in compression. The test is carried out for a specified exposure period with the specimen held under constant displacement using compact loading jigs. The compact nature of the jigs enables testing of several specimens in the test vessel simultaneously. Despite the apparent simplicity of the test, there are many factors that can influence the test results. The purpose of this standard is to establish a reliable methodology for conducting the tests to enhance repeatability and reproducibility of test data. The results of the tests can then be used with greater confidence to rank the performance of metals, the relative aggressiveness of environments, and to provide a basis for qualifying metals for service application. As such, the standard will be of particular benefit to materials and corrosion engineers in the oil and gas sector and to test houses providing critical data.

This standard was originally prepared in 2016 by Task Group 494, Four-Point Bend Test Method, which is administered by Specific Technology Group (STG) 32, Oil and Gas Production—Metallurgy. It is published under the auspices of STG 32.

In NACE standards, the terms *shall*, *must*, *should*, and *may* are used in accordance with the definitions of these terms in the *NACE Publications Style Manual*. The terms *shall* and *must* are used to state a requirement, and are considered mandatory. The term *should* is used to state something good and is recommended, but is not considered mandatory. The term *may* is used to state something considered optional.

NACE International
Standard Test Method

Four-Point Bend Testing of Materials for Oil and Gas Applications

Contents

FIRGUES

Section 1: General

1.1 This document provides guidelines for the use of four-point bend testing to evaluate the resistance of metals, including carbon steel, low alloy steels and corrosion resistant alloys (CRAs), to stress corrosion cracking and sulfide stress cracking. The emphasis in this document is on the methodology of the four-point bend test. The context of the test results for service application is the responsibility of the end-user and is discussed in NACE MR0175/ISO(1) 15156.[1-3]

Section 2: Principle

2.1 The four-point bend test is a constant displacement test that is performed by supporting a beam specimen on two loading rollers (bearing cylinders) and applying a load through two other loading rollers so that one face of the specimen is in tension (and uniformly stressed between the inner rollers) and the other is in compression. The stress at mid-thickness is zero and there will be significant gradients in stress through the thickness, this being most marked for thin specimens. As a consequence, cracks may initiate but then arrest, or their growth rate reduce. Hence, complete fracture may not always occur during the test exposure period. Important parameters are roller spacing, ratio between outer and inner span, specimen dimensions, width-to-thickness ratio, and roller diameter. Testing of as-welded specimens presents a particular challenge due to significant variations in root profile, surface roughness, extent of micro-cracks and degree of misalignment.

Section 3: Loading Jig Design

3.1 A loading jig similar to that shown in Figure 1 shall be used to apply a constant deflection to the specimen. The dimensions are often chosen so that A = H/4.

3.2 Specimens of thickness up to 5 mm present few problems for parent material specimens, as they can be easily accommodated in test vessels of modest size with typical dimensions for the loading jig of:

Spacing between inner rollers: 40-60 mm;
Spacing between outer rollers: 90-130 mm;
Roller diameter: 5-10 mm.

3.2.1 Spacing in this context refers to the distance from the center of one roller to the center of the other roller.

3.2.2 These dimensions are indicative. Other sizes may be adopted provided they are fit for purpose.

3.3 Thicker specimens, up to full wall thickness, are advisable for testing welded specimens. Here, there is a balance between minimizing the load by increasing the spacing between span supports and accommodating the increased size of the jig, with possible constraints associated with the size of the test vessel. This is an individual judgement.

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(1) International Organization for Standardization (ISO), Chemin de Blandonnet 8. Case Postale 401, 1214 Vermier, Geneva, Switzerland.

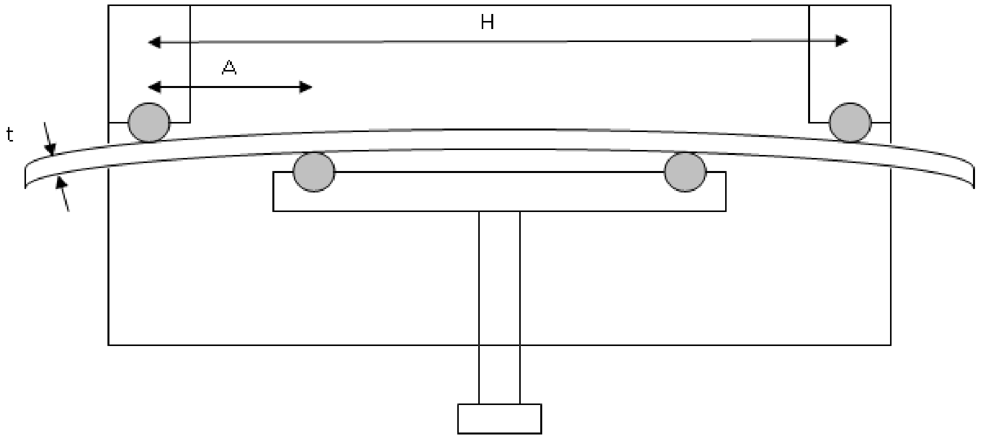

Figure 1: Schematic Illustration of Typical Four-Point Bend Loading Jig

NOTE: The width of the specimen should be machined with a tolerance of ±0.1 mm and the thickness machined to a tolerance of ±0.05 mm when using fully machined specimens. When testing with one surface in the as-welded state or as-processed there may be inherent local variations in thickness.

3.4 The specimen shall be electrically isolated from the loading jig in order to avoid undesirable galvanic and crevice corrosion. This is best achieved by the use of ceramic rollers, as these also satisfy the additional requirement that the rollers should not exhibit any yielding or creep during the test.

3.5 Friction between the rollers and the specimen should be minimized to limit the impact of frictional constraint on the stress distribution in the specimen. This is best achieved by the use of ceramic rollers that have a low friction contact surface, and may be further reduced if they are free to rotate while loading the test specimen.[4] In the absence of free rotation, there will be some effect of friction on the force required to achieve the required strain. However, provided the specimen is strain gauged and the frictional forces are not excessive, this will not impact on the strain in the central region of the specimen. Nevertheless, it could overstrain the specimen in the region local to the rollers with the possibility of cracks developing in the specimen in that region. The extent of overstraining for a particular loading jig can be assessed by strain gauging in that region for a typical test condition.

3.6 The material of construction of the loading jig shall be resistant to stress corrosion cracking in the test environment and the jig should be sufficiently rigid. Contamination of the solution with corrosion products from the jig material shall be minimised to avoid impacting on the test results. This may be achieved by the use of corrosion resistant alloys or by application of a coating to the jig. When testing carbon and low alloy steels with higher alloyed jigs, electrical bridging from corrosion products is a possibility and electrical resistance checks shall be made at test termination. Where electrical isolation is not used, then the material of construction of the jigs shall be similar to that of the specimens. For testing of carbon and low alloy steel specimens, adoption of low alloy steel jigs may be preferred to ensure an absence of galvanic interaction. In this case, a suitable inert coating may be applied to the jigs to minimize accumulation of corrosion products.

Section 4: Specimen Preparation

4.1 General

4.1.1 Four-point bend specimens shall be flat strips of metal of uniform rectangular cross section and uniform thickness, except in the case of testing welded specimens where testing is specified with one face in the as-welded condition, for which a non-uniform cross section is inherent, or when testing the inner surface of piping material in its original surface state (for which the surface would be concave) or outer surface of a piping material in its original surface state (for which the surface would be convex).

4.1.2 Identification marks or numbers shall be permanently inscribed on each end of the specimen. This is the region of lowest stress and the identification marks should therefore not initiate cracking.

4.1.3 Specimen preparation techniques that generate hydrogen at the specimen surface, e.g. electric discharge machining, should not be used on materials that are susceptible to hydrogen-induced damage. If the use of such techniques is necessary, a final grinding of the outer surfaces of the specimen shall be carried out to remove material containing retained hydrogen. The grinding shall be carried out as soon as possible to minimize the time available for the hydrogen to diffuse into the specimen from the outer surface. The thickness removed should reflect conservative evaluation of the effective hydrogen diffusivity in the material. For most corrosion resistant alloys, removal of 500 μm from each surface of the specimen is sufficient. Baking out of the hydrogen may also be considered, but only where this does not introduce changes in the material microstructure/microchemistry.

4.2 Parent Material Specimens

4.2.1 Parent material specimens shall be machined, avoiding sharp edges, from the pipe or plate in the longitudinal direction unless otherwise specified.

4.2.2 A typical four-point bend parent material specimen is shown in Figure 2(a).

4.2.3 The specimen width shall be at least 1.5 times the thickness of the specimen. Any deviation from this requirement, e.g. for very thick C-steel sections, requires demonstration that out-of-plane bending is not significant.

4.2.4 The specimen may be tested with the tensile test surface in its original surface state with no subsequent surface preparation. This recognizes that grinding always induces some change in the near-surface material properties and this may be undesirable. Otherwise, the surface of the specimen shall be prepared to a consistent repeatable finish as agreed with the end-user but usually with a Ra value ≤ 0.25 μm for any non-welded specimen. In the latter context, electropolishing or chemical pickling/passivation are not permitted for corrosion resistant alloys unless explicitly requested by the end-user. The test specimen shall be machined 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 test specimen shall be degreased with a suitable degreasing solution and rinsed with an appropriate solvent, such as acetone. Verification of the effectiveness of all cleaning procedures adopted in this standard shall be demonstrated; e.g. according to ASTM(2)-F21.[5]

NOTE: Preparation (machining) of environmental-cracking specimens from cold-hardened (i.e. cold-worked) stock, e.g., oilfield tubing and casing to ISO 13680 groups 2-4, can result in significant changes of strain in test surfaces before test strains (loads) are applied. This results from redistribution of residual stresses that may cause unintended under- or over- straining of test surfaces. Strain changes may be quantified by monitoring during machining. Currently, there is no established guidance on how such changes should be allowed-for during final specimen loading.

4.2.5 Deburring of the edges of the specimen may be undertaken by light manual grinding.

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(2) ASTM International (ASTM), 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959.

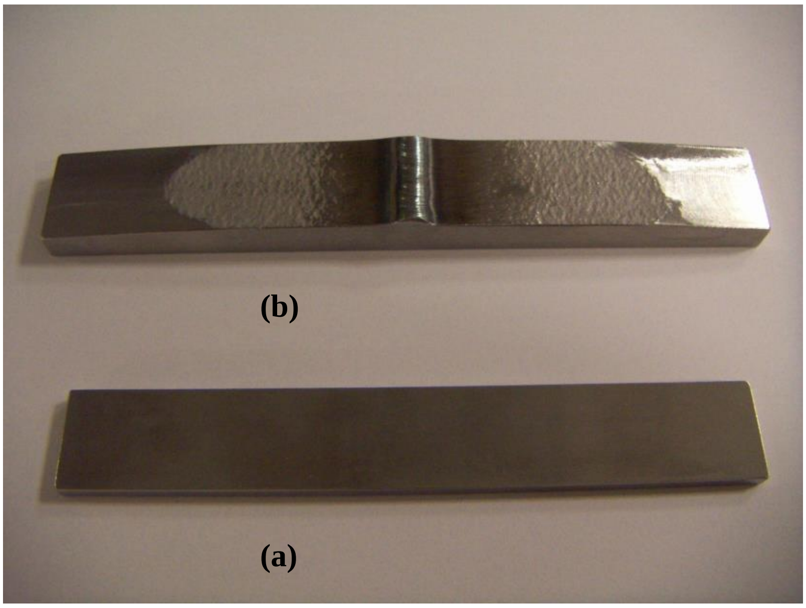

Figure 2: Typical Four-Point Bend Specimens: (a) Parent Material Specimen and (b) As-Welded Specimen

4.3 Welded Specimens

4.3.1 Unless specified otherwise, welded specimens shall be taken transverse to the weld, with the weld bead at the center of the specimen.

4.3.2 A typical four-point bend welded specimen is shown in Figure 2(b).

4.3.3 When testing with one surface in the as-welded state (in this context this means without further surface treatment by grinding), machining from one side only may often result in a variation in thickness on either side of the weld, because of misalignment of the sections during welding, and the extent of this shall be recorded. This variation in thickness will cause non-uniform straining of the specimen, but the impact should be less for thicker specimens. For this case, testing of near full-thickness specimens is preferred.

4.3.4 When testing specimens with one surface in the as-welded state, the locations in contact with the outer rollers should be machined flat to prevent high stress localization on the supports due to specimen curvature. Otherwise, cracking of the roller may occur.

4.3.5 For both fully machined and as-welded specimens, the specimen width shall be at least 1.5 times the thickness of the parent region of the specimen. Any deviation from this requirement, e.g. for very thick C-steel sections, requires demonstration that out-of-plane bending is not significant.

NOTE: In testing as-welded material with the weld-root intact, there will be inherent lateral curvature of the specimen. The effect of this will be to induce a higher strain toward the edges of the specimen, though the stress is not much changed. The effect becomes more pronounced the thicker the specimen, and hence to minimize the potential impact, a width-to-thickness ratio of 1.5:1 is recommended.[6]

4.3.6 The variation in thickness of the specimen due to tapering, misalignment and curvature (if the weld is machined from a pipe) shall be recorded.

4.3.7 When testing welds under fully machined conditions, the surface under tension should be as close as possible to the root surface as there may be hardness and microstructural variations through-thickness. In particular, the root pass shall be retained. Thus, 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, the location of the specimen surface in tension with respect to the actual pipe surface shall be noted and specimens cut in a consistent way. The surface shall be prepared to a consistent repeatable finish as agreed with end-user but usually with a Ra value ≤ 0.25 μm. The test specimen shall be fabricated carefully at an appropriate machining 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 shall be cleaned from the surface of the specimen using a suitable solvent and rinsed with acetone as per Paragraph 4.2.4.

4.3.8 Deburring of the edges of the specimen may be undertaken by light manual grinding.

4.4 Clad Product Specimens

4.4.1 When testing corrosion resistant alloy specimens from clad product, the carbon steel backing shall be completely removed by machining. This almost inevitably means that thin specimens will need to be used.

4.4.2 Complete removal of the carbon steel backing shall be checked using the copper sulfate test.[7]

4.4.3 For welded specimens, the weld root reinforcement (protrusion) shall be removed unless otherwise specified by the end-user. Removal of the reinforcement should be conducted in such a way as to minimize damage to the adjacent heat affected zone (HAZ)/parent regions, since the surface condition of these regions, in particular the heat tint, may influence the result.

Section 5: Strain Gauging

5.1 Strain gauging shall be used when the loading of the specimen is such that it could induce plastic deformation, as determined by prior derivation of material tensile properties. Guidance on strain gauging is given in Appendix A (nonmandatory).

5.2 For testing of parent material specimens at stresses where plastic deformation is induced, the strain gauge shall be attached to the calibration specimen at the center of the face in tension.

5.3 For testing of welded specimens, strain gauges shall be attached to the parent material in the center of the specimen symmetrically on either side of the weld metal as close as possible to the weld toes, but sufficiently far from them that the measured strain is not directly affected by any local stress/strain concentration, non-uniformity of the surface, or by the mechanical properties of the HAZ. A distance of the strain gauge sensors of between 3 mm and 5 mm from the weld toe is often adopted. The position of the strain gauges relative to the weld toe shall be recorded.

5.4 In strain gauging of as-welded material, attachment and subsequent removal of the gauges shall be undertaken in such a way so as to minimize changes in the surface state. Degreasing may be sufficient, with the solvents adopted having been validated as per Paragraph 4.2.4. Care shall be taken to minimize the area affected.

Section 6: Loading

6.1 Strain Level

6.1.1 The strain to be applied shall correspond to the required stress.

6.2 Setting the Total Strain Value

6.2.1 For parent material specimens, the objective is to achieve a specific value of strain at the center of the face of the specimen in tension.

6.2.2 The required deflection shall be measured at the center of the face of the specimen in tension. The deflection shall be measured using a suitable displacement monitor, such as a dial gauge or linear variable displacement transducer (LVDT) attached to the loading jig, as shown in Figure 3.

Figure 3: Loading Jig with Dial Gauge Attached for Measurement of Deflection

6.2.3 For applied stresses below the elastic limit, Equation (1) may be used to set the deflection,[8] y,

$$y = \frac{\left(3H^2 - 4A^2\right)\sigma}{12Et} \tag{1}$$

where σ is the required tensile stress, E is the modulus of elasticity, t is the specimen thickness, A is the distance between the inner and outer supports and H is the distance between the outer supports (see Figure 1). For carbon and low alloy steels and other materials that exhibit a distinct yield point in the tensile stress-strain curve, Equation (1) is then valid up to the yield point (the lower yield point in this case).

6.2.4 For materials that do not display a distinct yield point in the tensile stress-strain curve, the total strain (elastic and plastic) to give the required degree of plastic deformation (typically 0.2 % plastic strain) shall be identified using uniaxial stress-strain data (for example, see Figure 4).[9] The uniaxial data shall be based on three separate tensile tests (see ASTM E8[10]) using specimens prepared from the same heat treatment batch close to the location of source material and with same orientation from which the 4-point bend test specimens are obtained. The tensile specimens shall be in the form of parent material fully machined and ground to the surface finish specified in Paragraph 4.2.4.

6.2.5 The required deflection of the specimen is obtained when the magnitude of the longitudinal strain measured on the four point bend specimen corresponds to the total strain from the uniaxial test data (average of the three tests).[9]

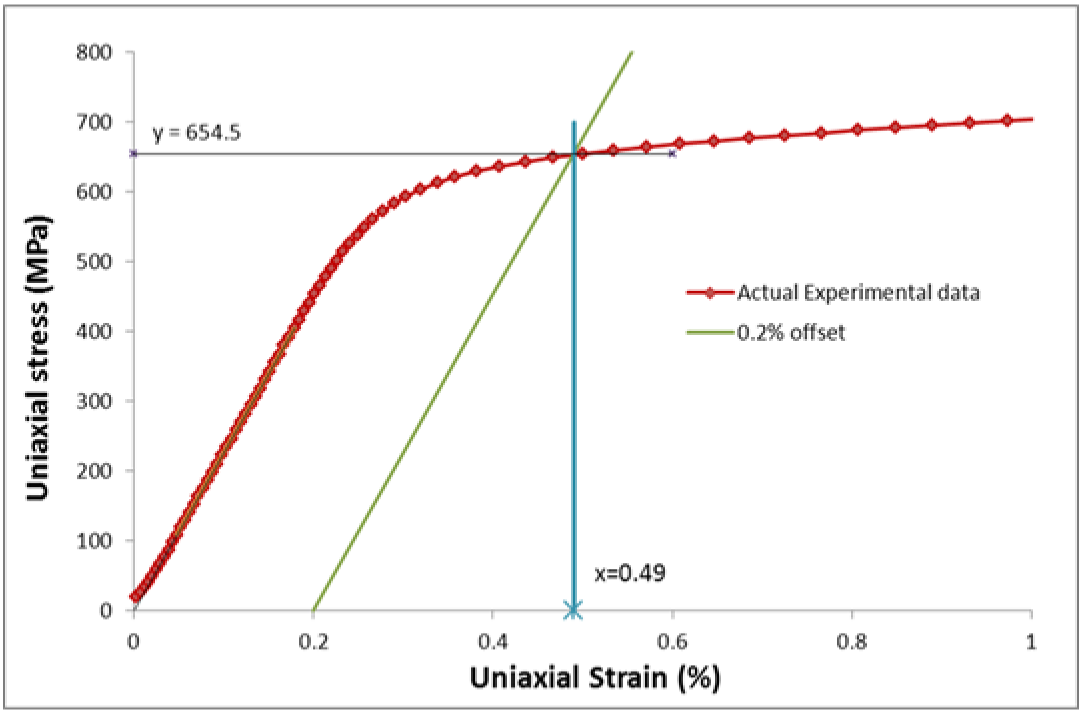

Figure 4: Typical Example of Uniaxial Stress-Strain Data for a Corrosion Resistant Alloy Showing Determination of Total Strain to Be Applied to Achieve 0.2% Plastic Strain

6.2.6 For calibration specimens, the deflection shall be measured using a suitable displacement monitor (Paragraph 6.2.2). Since the strain gauge on the calibration specimen is also positioned at the center of the face in tension, an adaptor shall be attached to the displacement monitor so that it bridges the strain gauge.

6.2.7 For welded specimens, the objective is to achieve the required level of strain in the parent material on at least one side of the weld. For fully machined welded specimens, a single calibration test may be sufficient to define the required deflection, but this should be validated. Because every as-welded specimen can be different, it is not possible to assign a specific deflection based on a particular calibration specimen. Each as-welded specimen shall be individually strain gauged. When loading welded specimens, the deflection is fixed when one of the strain gauges on either side of the weld first registers the required strain in the parent material.

6.2.8 In the case of dissimilar metal joints, the required strain shall be fixed in the lower strength parent material.

6.2.9 Any specimen strained beyond the intended level by more than 2% of the applied strain shall be discarded or tested in the overstrained condition.

6.3 Testing at Elevated Temperature

6.3.1 The mechanical properties of the material will change with temperature, the proof stress usually decreasing. This decrease is more marked for duplex stainless steel (DSS), compared with martensitic stainless steels and carbon and low alloy steels. Creep may also be a significant factor, e.g. for duplex stainless steels.

6.3.2 Uniaxial tensile tests (three tests) shall be conducted on reference specimens of the parent material at the test temperature in order to generate stress-strain data (see Appendix B [nonmandatory]).

6.3.3. For those alloys that exhibit a yield point, the test specimen shall be loaded at ambient temperature using equation (1) to a deflection that results in a stress corresponding to the high temperature yield point.

6.3.4 For alloys that do not exhibit a yield point, the total strain corresponding to 0.2 % plastic strain at the specified test temperature shall be determined from the stress-strain curve derived from uniaxial tensile tests at the test temperature (Paragraph 6.3.2). The test specimen shall then be loaded at ambient temperature until the longitudinal strain corresponds to the value of the total strain determined from the high temperature test. There will also be some differential expansion of specimen and jig that can result in some under-straining of the specimen, but the effect is insignificant.

6.3.5 Where creep at temperature is significant, a constant load rather than constant displacement method would be more conservative.

NOTE: Creep in four-point bend tests can be less than would be expected based on uniaxial tests because the stress gradient in the four-point bend test specimen provides a constraint to deformation.

Section 7: Test Environment

7.1 General

7.1.1 The test environment shall be a Fit-for-Purpose test solution representing a service application or shall conform to standard test solution such as Test Solutions A, B or C of NACE TM0177.[11] See Appendix C (nonmandatory).

NOTE: H_2S is highly toxic and must be handled with caution. See Appendix D (nonmandatory).

7.1.2 The test vessel material shall not lead to contamination of the test environment for the specified test conditions and shall not compromise safety.

7.1.3 Test vessels shall be sized to maintain the test solution volume within the specified limits relative to the exposed surface area of the test specimen to standardize the drift of pH with time (see NACE TM0177).[11]

7.1.4 Methods of deaeration and transfer of test solution to the test vessel shall be used that result in a sufficiently deaerated test solution. The oxygen concentration in the test solution shall be maintained below 10 ppb when testing corrosion resistant alloys. In tests using non-metallic vessels, a nitrogen cabinet may be used to avoid oxygen ingress through the seals or through the containment vessel or connections. When testing low-alloy steels, the oxygen concentration shall be less than 50 ppb unless the strength level is greater than or equal to 80 ksi (552 MPa) for which the oxygen concentration shall be maintained below 10 ppb.

7.1.5 Monitoring of the oxygen concentration in each test is not necessary. Instead, a separate test shall be conducted using the same apparatus and procedure, but with the oxygen concentration monitored, to demonstrate that the methodology achieves the required level of oxygen. Evidence showing achievement of the required oxygen level shall be documented.

7.1.6 The test temperature shall be maintained within ± 3 °C of the target value unless otherwise specified.

7.1.7 When calculating the total pressure in tests at elevated temperature, due allowance shall be made for the partial pressure of water vapor.

7.1.8 When calculating the composition of the test gas, the partial pressure of each gas should be used rather than its fugacity, as this approach is generally conservative. In cases where this is deemed not appropriate, for instance, when simulating a specific high pressure service environment, the alternative approach used shall be agreed and documented in detail.

7.1.9 All chemicals used shall be reagent grade or chemically pure (99.5 % minimum purity) chemicals. The test water shall be distilled or deionized water with a quality equal to or greater than ASTM Type IV (ASTM D 1193-6).[12]

7.2 pH Adjustment

7.2.1 The solution pH may be fixed either by the specified water chemistry, temperature and partial pressures of CO_2 and H_2S or it may be adjusted by addition of appropriate amounts of acid/alkali/buffer as specified in Appendix C for both CRAs and carbon steels.

7.2.2 Adjustment of solution pH, where permitted, shall be carried out at ambient temperature and ambient pressure after saturation with the test gas, e.g. H_2S, H_2S/CO_2 or other gas mixture, or pure CO_2, unless this is deemed to inadvertently affect the material to be tested, or risk ingress of oxygen, in which case any alternative approach used shall be documented.

NOTE: The pH measured at ambient temperature and pressure may not be the same as that at the test temperature and pressure in the presence of the appropriate test gas and due account should be taken of this when deciding what “control pH” to adjust to at ambient temperature and pressure.

7.2.3 A calibrated pH meter shall be used to measure the solution pH as described in Paragraph 7.2.2.

7.2.4 When using Test Solutions A or B of NACE TM0177, intentional adjustment of pH during the test is prohibited.

7.3 pH Control

7.3.1 After the test, the pH shall be measured at ambient temperature and pressure in the test solution saturated with the test gas mixture (for ambient pressure tests) or under pure CO_2 (for elevated pressure tests) and the result recorded (unless deemed inappropriate as outlined in Paragraph 7.2.2).

7.3.2 Where testing is performed in one of the standard solutions given in TM0177 for their specified use, the respective rules for pH control and measurement in TM0177 apply.

7.3.3 If Paragraph 7.3.2 does not apply, the variation of the test pH from the control point shall not exceed ±0.2 pH units.

Section 8: Procedure for Four-Point Bend Testing

8.1 Determine the required deflection or strain for the specimen, as described in Section 6.

8.2 Before testing, the test specimen shall be degreased with a suitable degreasing solution and rinsed with an appropriate solvent, such as acetone, then stored in a dessicator. The adequacy of the degreasing procedure shall be validated, e.g. according to ASTM F21.[5]

8.3 Place the specimen in the loading jig and load the specimen to the required deflection or strain. Check for any strain relaxation. This may be done as follows, though for one specimen of the same batch of material only:

For elastically loaded alloys, measure the strain after 1 h. If relaxation has occurred, adjust the deflection and repeat after 1 h. In all other cases, measure the strain at 5 min intervals and adjust the deflection until the strain is constant for at least 30 min. If it has not changed, the test can be started. Otherwise, the deflection shall be adjusted to attain the required strain and checked after 1 h to ensure no significant relaxation has occurred.

In all cases, the extent of initial stress relaxation together with description of the adjustments made shall be reported.

8.4 Place the loading jig in the test vessel, then seal the lid and ensure there are no leaks.

8.5 The test solution shall be added to the test vessel in such a way as to meet the requirements of Paragraph 7.1.4. Paragraphs 8.6-8.9 provide an often suitable, though not exclusive, methodology.

8.6 Place the test solution in a separate reservoir and deaerate by purging with a suitable low oxygen purging gas (see Paragraphs 7.14 and 7.15). Purging with CO_2 should be considered if there is a possibility of precipitation of sparingly soluble salts that have reduced solubility with increasing pH.

NOTE 1: The time to achieve a steady state concentration of dissolved gas will depend on the size of the gas bubbles, the period in contact with the water (thus the height of the water relative to the bottom of the bubbler) and the flow rate. At a gas flow rate of 0.1 L/min, a period of 20 h is required for a 20 L solution and for 10 L (shorter height), a period of 12 h is required.[13]

NOTE 2: Deaerating with an inert gas will cause purging of dissolved CO_2 and loss of bicarbonate ions, as the ions are in equilibrium with dissolved CO_2. The pH will increase with purging as the sodium ions added as sodium bicarbonate are now balanced by hydroxyl ions, with the increase in pH dependent on the concentration of sodium bicarbonate added. Upon charging with the test gas, equilibrium will be restored and the sodium hydroxide will reconvert to sodium bicarbonate (as confirmed by pH measurement after testing). However, during the purging period, any salt whose solubility is diminished with increasing pH may precipitate.

8.7 The test vessel and connecting tubes shall be deaerated prior to injection of the solution from the reservoir with the method chosen to ensure no impact on the test specimen.

NOTE: For corrosion resistant alloys, it can be sufficient to connect the gas outlet from the reservoir to the gas inlet of the test vessel. The outlet gas from the test vessel should be passed through an outlet trap (e.g. Dreschel bottle) to prevent oxygen ingress. For carbon steels, carry-over of water with some oxygen initially present in the gas stream could cause corrosion, and this method is not appropriate.

8.8 Pump the solution into the test vessel using the pressure of the purging gas.

8.9 Saturate the solution with the test gas using a flow rate and bubble size appropriate to attaining saturation in an optimum timescale. For most cases a flow rate of 0.1 L/min and 1 h/L of test solution is sufficient to achieve near saturation, assuming at least a 5L test solution. Alternative charging rates may be adopted for larger vessels, for example, but evidence showing attainment of saturation shall be documented. The gas concentration in solution shall be maintained at the required level during the test. This may be achieved by continuous or periodic replenishment as described, for example, in TM0177. For CRAs, the depletion of H_2S may be sufficiently small that replenishment is not required during the test, but evidence to support this approach shall be provided.

NOTE: As a reference, for testing with saturated H_2S at ambient temperature and pressure, the saturation concentration should be in excess of 2,300 mg/L when using Test Solution A of TM0177.

8.10 For testing at elevated temperature, bring the test vessel/autoclave to the test temperature and then increase the gas partial pressures to the required level as appropriate. Alternatively, set the gas partial pressures at ambient temperature to attain the desired partial pressures at the test temperature. The methodology for calculating the partial pressure at temperature shall be documented.

8.11 Upon attainment of steady conditions, expose for the required period (typically 30 days).

8.12 At the end of the exposure period, dissolved H_2S shall be removed. This may be achieved by purging the test solution with nitrogen. For elevated temperature tests, purging should be undertaken at the test temperature to minimize the possibility of inducing sulfide stress cracking at a lower temperature while cooling.

NOTE: For testing of carbon steels with a sealed-in system with no replenishment of test gas, depletion of H_2S may occur during the course of the test. Depending on the requirements of the end user, it may be necessary to measure the concentration of dissolved H_2S at the end of the test.

8.13 Caution shall be exercised in opening the vessel, as residual H_2S could remain.

8.14 Subsequently remove the specimens, rinse with water and dry with acetone.

8.15 Photograph the specimen, if required by the end-user, prior to removal of corrosion product.

8.16 If necessary, remove any corrosion product; for example, using an inhibited HCl solution.

NOTE: Appropriate safety measures should be taken, since H_2S may be released when the corrosion products are being dissolved.

Section 9: Failure Appraisal

9.1 Carbon Steel

9.1.1 Test specimens shall be evaluated in the tensile stressed region between the inner loading rollers for any evidence of cracking, including surface breaking cracks, sub-surface hydrogen cracks, sub-surface/surface breaking stress oriented hydrogen induced cracking (SOHIC) and soft zone cracking (SZC).

9.1.2 The following methods should be used, with increasingly more detailed examination adopted where no surface cracking is observed:

i. Initial visual examination at 10x magnification
ii. Non-destructive assessment of the presence of cracks; using, for example, magnetic particle inspection (MPI) or liquid penetrant testing for surface cracks on the stressed test face, or Ultrasonic Testing
iii. Sectioning of the specimens at any suspicious features noted in Steps (i) and (ii);
iv. Otherwise, where no surface cracks are detected, undertake longitudinal sectioning at two locations (typically at 1/3 and 2/3 of width) followed by metallographic preparation and examination in the unetched condition at 100x magnification of cut faces. The size and location of any cracks shall be confirmed in the etched condition.

9.1.3 All cracks identified shall be reported, identifying the type of crack and location.

NOTE: Specifying the location of cracking is important because enhanced stress and deformation along the specimen edge may induce cracking on the specimen edge that might not otherwise occur. Similarly, cracking may occur preferentially in the vicinity of the rollers because of an elevated local stress and strain compared to that in the stressed region between the rollers.

9.1.4 An unstressed and unexposed reference specimen may be evaluated for any evidence of cracking as per Paragraph 9.1.2.

NOTE: Cracks or crack-like flaws may be generated in the material during processing/welding and could be confused with cracks generated during exposure testing.

9.1.5 The visual observation of corrosion pits or other notable features shall be recorded.

NOTE: In the absence of cracks, consideration should be given to whether corrosion pits could continue to propagate and transform to cracks at longer test duration. To assess that possibility, the maximum pit depth should be determined (see ISO 11463.)[14]

9.2 Corrosion Resistant Alloys

9.2.1 Examine specimens in the tensile stressed region between the inner loading rollers using low powered microscopy to at least 10x magnification. Photographic evidence of any cracking shall be recorded.

NOTE: Specifying the location of cracking is important because enhanced stress and deformation along the specimen edge may induce cracking on the specimen edge that might not otherwise occur. Similarly, cracking may occur preferentially in the vicinity of the rollers because of an elevated local stress and strain compared to that in the stressed region between the rollers.

9.2.2 Visual/low powered microscopic examination may be complemented by dye penetrant examination (DPE) or fluorescent dye penetrant examination (FDPE).

9.2.3 The visual observation of pits or other notable features shall be recorded.

NOTE: In the absence of cracks, consideration should be given to whether corrosion pits could continue to propagate and transform to cracks at longer test duration. To assess that possibility, the maximum pit depth should be determined (see ISO 11463.)[14]

9.2.4 Where no surface cracks are visible, undertake longitudinal sectioning at two locations (typically at 1/3 and 2/3 of width) and conduct metallographic examination at up to 100x magnification.

9.2.5 An unstressed and unexposed reference specimen may be evaluated for any evidence of cracking as per Paragraphs 9.2.2 and 9.2.3.

NOTE: Cracks or crack-like flaws may be generated in the material during processing/welding and could be confused with cracks generated during exposure testing.

9.2.6 The visual observation of pits or other notable features shall be recorded.

NOTE: In the absence of cracks, consideration should be given to whether corrosion pits could continue to propagate and transform to cracks at longer test duration. To assess that possibility, the maximum pit depth should be determined.[14]

Section 10: Test Report

As a minimum, the test report shall include the following information, where applicable:

a) Full description of the test material, including heat number, heat treatment lot, mechanical properties, composition and structural condition, type of product, welding parameters (where known);
b) The target stress and applied deflection;
c) Location from which specimen was removed; orientation of specimen, curvature (if any), dimensions (including any non-uniformity of thickness), commentary on root profile (for as-welded specimens), surface preparation, photographs (if requested);
d) Four-point bend test setup data;
e) Strain gauging procedure;
f) Loading procedure;
g) Environment composition, including initial and final pH and any pH adjustment made, gas composition, test temperature and exposure time;
h) Method used for detecting cracks;
i) Presence and location of cracks on specimens, observed crack depth and path (where determined), photographic evidence of cracking (if any);
j) Presence and location of any pits on specimens with photographic evidence of pitting (if any); maximum pit depth when measured.

References

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15. ISO 6892-1 (latest revision), “Metallic materials - Tensile testing - Part 1. Method of test at room temperature” (Geneva, Switzerland: ISO).

16. ISO 6892-2 (latest revision), “Metallic materials - Tensile testing - Part 2. Method of test at elevated temperature” (Geneva, Switzerland: ISO).

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(3) National Physical Laboratory (NPL), Hampton Road, Teddington, Middlesex, TW11 0LW, U.K.

(4) British Society for Strain Measurement (BSSM), PO Box 839, Flitwick, Bedford, MK45 9DU, U.K.

(5) American Chemistry Council (ACC) (formerly known as the Manufacturing Chemists Association and then as the Chemical Manufacturers Association), 700 Second St. NE, Washington, DC 20002.

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(6) European Federation of Corrosion (EFC), 1 Carlton House Terrace, London, SW1Y 5DB, U.K.

(7) The Institute of Materials, Minerals and Mining, 297 Euston Road, London NW1 3AQ, U.K.

Appendix A
Procedure for Strain Gauging and Determining Uniaxial Stress-Strain Calibration Curve
(Nonmandatory)

This appendix is considered nonmandatory, although it may contain mandatory language. It is intended only to provide supplementary information or guidance. The user of this standard is not required to follow, but may choose to follow, any or all of the provisions herein.

A1 Preparation of uniaxial tensile test reference specimens for determining total strain for 4 point bend testing

The strain on the tensile test specimen can be measured by extensometry or by strain gauging. To account for the possibility of bending due to imperfect alignment, measurement should be made on opposite sides of the specimen and the average strain used when plotting the stress-strain curve. Guidelines for use of extensometry are given in ISO 6892 Parts 1 and 2.[15,16] Here, some additional notes to guide the application of strain gauges are given as this also relates to the methodology for four-point bend testing.

The strain gauges, cabling, adhesive and consumables selected shall be rated for the required adhesive cure and test temperatures.

A useful reference for strain gauge installation is the British Society for Strain Measurement (BSSM) code of practice CP1.[17]

- Lightly abrade the surface using 400 grade silicon carbide paper
- Clean the surface to remove residue of grinding process with suitable solvent such as isopropanol
- Use a mild acidic fluid and neutralizing agent to make the surface chemically inert
- Lightly mark the position of the strain gauge using a fine tip hard lead pencil or ballpoint pen
- Place the strain gauge on low tack tape and position over the marked lines
- Apply adhesive as per the manufacturer's recommendations (this step should be done within 20 minutes of the abrasion stage)
- Use a spring clamp to hold the specimen in place and apply a light clamping force during cure
- Cure and post cure the sample to the manufacturer's recommendations, typically the post cure temperature is 30-40 °C above the operating temperature
- Remove all tape and consumables
- Check gauge installation and record all details of strain gauge and installation in log book or on worksheets

A2. Testing of uniaxial reference specimens at test temperature using strain gauges or extensometry

- Attach strain gauges or extensometers on opposite sides of the tensile test specimen
- Position specimen centrally in jig (wedge action for flat dog-bone specimens);
- Place thermocouple onto specimen surface
- Check strain measurement devices are functioning correctly, balance load & displacement
- Heat the specimen, in temperature controlled chamber, to required temperature using thermocouple on specimen as reference
- Once the specimen is at temperature allow it to stabilise for 30 minutes
- Move crosshead until it just starts to take load, re-balance displacement
- Initiate strain monitoring equipment
- Load specimen at an appropriate rate (e.g. 1 mm/min; equivalent to a strain rate of 6.7×10^{-4} s^{-1} for a 25 mm gauge length, though note the actual strain rate will depend on machine stiffness)

NOTE: In general, the thickness of the strain gauge and adhesive can be ignored in relation to their impact on the measured strain, but if the specimen is particularly thin, the increased distance of the measurement probe from the neutral axis cannot be neglected and an appropriate adjustment should be made.

Appendix B
Modulus Calculation
(Nonmandatory)

This appendix is considered nonmandatory, although it may contain mandatory language. It is intended only to provide supplementary information or guidance. The user of this standard is not required to follow, but may choose to follow, any or all of the provisions herein.

There is value in checking the stiffness of the loading frame in four-point bend testing. This can be done by measuring the load-strain curve, calculating the modulus and ensuring that this concurs with literature data for the test material at the test temperature. Deviation from literature data would indicate that the stiffness of the jig was inadequate or that some other aspect of the test methodology was insufficiently robust.

The modulus should be estimated from the linear region of the load-strain data using Equation (B1):

$$E = \sigma / \varepsilon \tag{B1}$$

where E is the modulus of elasticity, ε is the tensile strain, and σ is the tensile stress which is given by Equation (B2):

$$\sigma = \frac{3d_1W}{wt^2} \tag{B2}$$

where d_1 is half the difference in distance between the inner and outer rollers, W is the applied load, w is the specimen width and t is the specimen thickness.

Appendix C
Specification of Solution Chemistry and its Control for Different Standards
(Nonmandatory)

This appendix is considered nonmandatory, although it may contain mandatory language. It is intended only to provide supplementary information or guidance. The user of this standard is not required to follow, but may choose to follow, any or all of the provisions herein.

Standard	**Section**	**Type**	**Test Pressure**	**Temp. (°C)**	**Solution Chemistry**	**Buffer**	**Test Gas**		**pH**						**Comments**
							H_2S	CO_2	Test	Initial	Interim	Final	Tolerance	Adjust	
		CRA's													
NACE MR0175/ ISO 15156	B.3.5.2	1	Ambient or greater	Any	Synthetic produced water that simulates the chloride and bicarbonate concentrations of intended service	None or HCO3	Service partial pressure	Service partial pressure	in-situ	Measure at ambient temperature and pressure under the test gas or pure CO2	Not required	Measure at ambient temp. and pressure under the test gas or pure CO2	None	No	
	B.3.5.2	2	Ambient	60 max	Chloride at concentration of intended service	HCO3 added to achieve required pH	Service partial pressure	Balance to 1 bara	Specified	Measure + adjust	Measure + adjust	Measure	±0.2	Yes	

	B.3.5.4	3a	Ambient	24±3	Chloride at concentration of intended service	4g/l NaAC - HCl	Service partial pressure	Balance to 1 bara	Specified	Measure + adjust	Measure + adjust	Measure	±0.2	Yes	
	B.3.5.4	3b	Ambient	24±3°	Chloride at concentration of intended service	0.4g/l NaAC - HCl	Service partial pressure	Balance to 1 bara	Specified	Measure + adjust	Measure + adjust	Measure	±0.2	Yes	Same solution as NACE TM0177-Solution C
NACE TM0177	6.1	A	Ambient	Any	5.0 wt% Sodium chloride + 0.5 wt% glacial acetic acid	None	1 bara or service partial pressure	Balance to 1 bar	Prepared = 2.6 - 2.8	≤3	≤4	≤4	NA	No	
Standard	**Section**	**Type**	**Test Pressure**	**Temp. (°C)**	**Solution Chemistry**	**Buffer**	**Test Gas**		**pH**						**Comments**
							H_2S	CO_2	Test	Initial	Interim	Final	Tolerance	Adjust	
NACE TM0177	6.3	C	Ambient	24±3	Chloride at concentration of intended service	0.4g/l NaAC – HCl/NaOH	1 bara or service partial pressure	Balance to 1 bar	Specified	Measure + adjust	Measure + adjust	Measure	±0.2	Yes	Intended for martensitic stainless steels (Same solution as 3b)

Carbon Steels															
NACE TM0177	6.1	A	Ambient	Any	5.0 wt% Sodium chloride + 0.5 wt% glacial acetic acid	None	1 bara or service partial pressure	Balance to 1 bar	Prepared = 2.6 - 2.8	≤3	≤4	≤4	NA	No	
NACE TM0177	6.2	B	Ambient	Any	5.0 wt% Sodium chloride + 2.5 wt% glacial acetic acid	0.41 wt% sodium acetate	1 bara or service partial pressure	Balance to 1 bar	Prepared = 3.4 - 3.6	≤4	≤4	≤4	NA	No	
NACE MR0175/ ISO 15156	B.3		Ambient	24± 3	5 wt% sodium chloride	0.4 wt% sodium acetate + HCl/NaOH	1 bara or service partial pressure	Balance to 1 bar	Specified	Measure	Measure	Measure	±0.1	Yes	
EFC 16	A.3		Ambient	23± 2	50 g/L sodium chloride	4g/L sodium acetate + HCl/NaOH	1 bara or service partial pressure	Balance to 1 bar	Specified	Measure	Measure	Measure	±0.1	Yes	

Appendix D
Safety Considerations in Handling H_2S Toxicity
(Nonmandatory)

This appendix is considered nonmandatory, although it may contain mandatory language. It is intended only to provide supplementary information or guidance. The user of this standard is not required to follow, but may choose to follow, any or all of the provisions herein.

H_2S is perhaps responsible for more industrial poisoning accidents than is any other single chemical. A number of these accidents have been fatal. H_2S must be handled with caution and any experiments using it must be planned carefully. The OSHA(8) maximum allowable concentration of H_2S in the air for an eight-hour work day is 20 mg/L, well above the level detectable by smell.[19] However, the olfactory nerves can become deadened to the odor after exposure of 2 to 15 minutes, depending on concentration, so that odor is not a reliable alarm system.

Briefly, the following are some of the human physiological reactions to various concentrations of H_2S. Exposure to concentrations in the range of 150 to 200 mg/L for prolonged periods may cause edema of the lungs. Nausea, stomach distress, belching, coughing, headache, dizziness, and blistering are symptoms of poisoning in this range of concentration. Pulmonary complications, such as pneumonia, are strong possibilities from such subacute exposure. At 500 mg/L, unconsciousness may occur in less than 15 minutes, and death within 30 minutes. At concentrations above 1,000 mg/L, a single inhalation may result in instantaneous unconsciousness, complete respiratory failure, cardiac arrest, and death.

Additional information on the toxicity of H_2S can be obtained from the Chemical Safety Data Sheet SD-36[20] and from *Dangerous Properties of Industrial Materials*.[21]

Fire and Explosion Hazards

H_2S is a flammable gas and yields poisonous sulfur dioxide (SO_2) as a combustion product. In addition, its explosive limits range from 4 to 46% in air. Appropriate precautions shall be taken to prevent these hazards from developing.

Safety Procedures During Test

All tests shall be performed in a hood with adequate ventilation to exhaust all of the H_2S. The H_2S flow rates during the test should be kept low to minimize the quantity exhausted. A 10% caustic absorbent solution for effluent gas can be used to further minimize the quantity of H_2S gas exhausted. This caustic solution needs periodic replenishing. Provision shall be made to prevent backflow of the caustic solution into the test vessel if the H_2S flow is interrupted. Suitable safety equipment shall be used when working with H_2S.

Because the downstream working pressure frequently rises as corrosion products, debris, etc., accumulate and interfere with regulation at low flow rates, particular attention should be given to the output pressure on the pressure regulators. Gas cylinders shall be securely fastened to prevent tipping and breaking of the cylinder head. Because H_2S is in liquid form in the cylinders, the high-pressure gauge must be checked frequently, because relatively little time elapses after the last liquid evaporates and the pressure drops from 1.7 MPa (250 psig) to atmospheric pressure. The cylinder shall be replaced by the time it reaches 0.5 to 0.7 MPa (75 to 100 psig) because the regulator control may become erratic. Flow shall not be allowed to stop without closing a valve or disconnecting the tubing at the test vessel, because the test solution continues to absorb H_2S and move upstream into the flowline, regulator, and even the cylinder. A check valve in the line should prevent the problem if the valve works properly. However, if such an accident occurs, the remaining H_2S should be vented as rapidly and safely as possible and the manufacturer notified so that the cylinder can be given special attention.

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(8) Occupational Safety and Health Administration (OSHA), U.S. Department of Labor, 200 Constitution Ave. NW, Washington, DC 20210.

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