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

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

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 face of the specimen to be tested is stressed in tension and the reverse face 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 laboratories providing critical data.

Scope

This document provides technical requirements for the use of four-point bend testing to evaluate the resistance of metals, including carbon steels, low alloy steels, and corrosion resistant alloys (CRAs), to stress corrosion cracking (SCC) and sulfide stress cracking (SSC). The test is carried out for a specified exposure period with the specimen held under constant displacement using compact loading jigs.

Rationale

The standard is revised as required by the AMPP 5-year review policy. Revisions are mainly focused on specimen loading by Deflection and Strain Gauge Methods. The revisions provide more clarifications and specifications on how to load a four-point bend specimen to the target stress level.

In AMPP standards, the terms *shall* and *must* are used to state requirements and are considered mandatory. The term *should* is used to state something that is recommended, but is not considered mandatory. The term *may* is used to state something considered optional.

Section 1: General

This document provides technical requirements for the use of four-point bend testing to evaluate the resistance of metals, including carbon steels, low alloy steels, and corrosion resistant alloys (CRAs), to stress corrosion cracking (SCC) and sulfide stress cracking (SSC). The emphasis in this document is on the methodology of the four-point bend test. The context of the test results for service application and applicability of ANSI[(1)]/NACE MR0175/ISO[(2)] 15156[1-3] is the responsibility of the service purchaser/end-user.

Although this test method is intended for SCC or SSC testing, other types of cracking, e.g., hydrogen-induced cracking (HIC), stepwise cracking (SWC), stress-oriented hydrogen-induced cracking (SOHIC), may also be observed.

The default parameters defined by this document are standard requirements.

Section 2: Terms, Definitions and Abbreviations

For the purposes of this document, the following terms, definitions, and abbreviated terms apply.

As-received Parent Material Specimen: The specimen taken from the part of a material sample in the original condition of interest (manufacturing form, post-service, etc.).

(1) American National Standards Institute (ANSI), 25 West 43rd St., 4th Floor, New York, NY 10036, www.ansi.org.
(2) International Organization for Standardization (ISO), Chemin de Blandonnet 8. Case Postale 401, 1214 Vernier, Geneva, Switzerland, www.iso.org.

As-welded Specimen: The specimen taken from a weldment, in which the test face contains the root or cap of weld without machining and/or grinding.

Longitudinal: The orientation along the length of specimen, when referring to the specimen, not to the processing/geometry of the product that has been manufactured. For the product to be tested, it signifies that the lengthwise axis of the specimen is parallel to the direction of the greatest extension of the material during rolling or forging.

Machined Parent Material Specimen: The fully machined prismatic specimen taken from the part of a material sample in the original manufacturing form.

Machined Weldment Specimen: The prismatic specimen taken from a weldment, in which the test face has been fully machined or ground, i.e., the test face is not an original surface of the welded sample.

Transverse: The orientation along the width of the specimens, vertical orientation to the specimen lengthwise axis when referring to the specimen, not to the processing/geometry of the product that has been manufactured. For the product to be tested, it signifies that the lengthwise axis of the specimen is right angles to the direction of the greatest extension of the steel during rolling or forging.

Section 3: Principle

The four-point bend test is a constant displacement test that is performed by supporting a beam specimen and applying a bending load through four loading rollers such that the test face of the specimen is in tension (and uniformly stressed between the inner rollers) and the reverse face is in compression, with a neutral plane (zero stress) close to mid-thickness. There will be significant gradients in stress through the thickness, this being most marked for thin specimens, and consequently individual 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 or cap profile, surface roughness, extent of micro-cracks, and degree of misalignment.

Section 4: Loading Jig Design and Test Specimen Dimensions

4.1 A loading jig similar to that as shown in Figure 1 shall be used to apply a constant deflection to the test specimen.

4.2 Typical dimensions of the loading jig are as follows:

Spacing between inner rollers, H-2A: 40-60 mm;
Spacing between outer rollers (i.e., span), H: 90-130 mm;
Mid-span (H/2): 45-65 mm;
Roller diameter: 5-10 mm.

NOTE 1: The dimensions are often chosen so that A = H/4.

NOTE 2: Spacing in this context refers to the distance from the center of one roller to the center of the other roller.

4.2.1 Jig dimensions specified above are indicative. Other sizes may be adopted, provided that they are fit for purpose.

NOTE: Other loading jig designs are acceptable if the jig dimension ratios illustrated in Figure 1 are maintained.

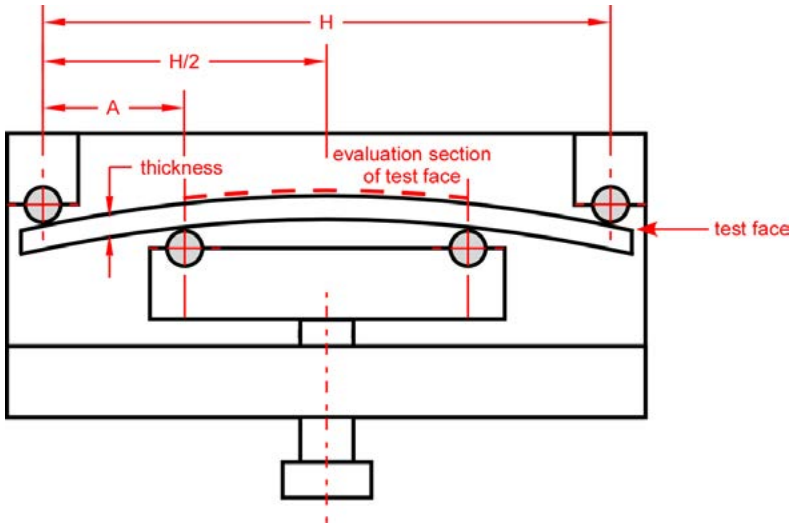

Figure 1: Schematic Illustration of Typical Four-Point Bend Loading Jig

4.3 The specimen width should be ≥1.5 times the thickness of the specimen.

NOTE: Test specimens of 4-10 mm thickness present few problems, as they can be easily accommodated in test vessels of modest size.

4.4 Typical test specimen dimensions are shown in Table 1.

Table 1
Typical Specimen Dimensions (Nonmandatory)

Specimen Type	Length	Width	Thickness
Parent Material (Machined or As-received)	102 - 140 mm (4.0 – 5.5 in)	12-20 mm (0.5 - 0.8 in)	4-6 mm (0.16 – 0.24 in)
Machined Weldment	102 – 140 mm (4.0 – 5.5 in)	12-20 mm (0.5 - 0.8 in)	4-6 mm (0.16 – 0.24 in)
As-welded	127 - 152 mm (5.0 – 6.0 in)	15-25 mm (0.6 – 1.0 in)	5-10 mm (0.20 – 0.40 in)*

* Thickness of the parent portion of the specimen

NOTE: The specimen dimensions noted in Table 1 are indicative. Other sizes may be adopted, provided that they are fit for purpose. Keeping specimen proportionality to dimensions listed in Table 1 is recommended in the case of alternate size specimens.

4.4.1 The uniformity of dimensions on a single specimen or for multiple similar specimens shall be machined to a width tolerance of ±0.1 mm and a thickness tolerance of ±0.05 mm when using fully machined specimens. When testing the test face in the As-welded or As-received condition, there may be inherent local variations in thickness.

NOTE: Using thick specimens allows more even loading and can improve the reliability of the test method.

4.4.2 The specimen (straight or curved) loaded to the desired applied stress shall not be in contact with the jig. This shall be verified prior to specimen loading.

4.5 The material of construction of the loading jig shall be resistant to both corrosion and environmentally assisted cracking damage mechanisms in the test environment and the jig should be sufficiently rigid. Contamination of the solution with corrosion products from the jig material shall be minimized to avoid impacting on the test results. This can be achieved by using corrosion resistant alloys (CRAs), by the application of an inert coating to the jig, or by using more volume of solution to achieve the required minimum solution-volume to specimen-surface-area ratio.

4.6 The loading jig shall be electrically isolated from the test specimen, in order to eliminate possible galvanic effects. Electrical isolation is best achieved by using ceramic rollers, as these also satisfy the additional requirement that the rollers should not exhibit any yielding or creep during the test.

4.7 Friction between the rollers and the test specimen should be minimized to limit the impact of frictional constraint on the stress distribution in the specimen. This is best achieved by using ceramic rollers that have a low friction on the contacted surface and can further reduce the friction 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, friction effects will not impact on the strain in the central region of the test specimen. Nevertheless, it could overstrain the test specimen in the region local to the inner rollers with the possibility of cracks developing in the test specimen in that region. The extent of overstraining for a particular loading jig can be assessed by strain gauging in that region.

Section 5: Specimen Preparation

5.1 General

5.1.1 Four-point bend specimens shall be flat strips of metal with uniform rectangular cross section and uniform thickness, except in the case of testing As-welded specimens (for which a non-uniform cross section is inherent), 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).

5.1.2 Specimen identification marks (e.g., stencil marks) should be located at both ends of the test specimen, outside of the outer loading rollers. This is the region of lowest stress, and the identification marks will therefore not initiate cracking.

NOTE: Specimen preparation techniques can potentially generate hydrogen at the test specimen surface(s) by, e.g., electrical discharge machining. A final grinding of each test specimen surface is recommended to remove material containing retained hydrogen. The thickness removed should reflect the greatest effective hydrogen diffusivity in the material. For most corrosion resistant alloys, removal of 500 μm from each surface of the test specimen is sufficient. Baking specimens to remove potential retained hydrogen may also be considered, but only for the case that this baking treatment does not introduce changes in the material microstructure/microchemistry.

5.1.3 For Machined specimens (Parent Material or Weldment), the average surface roughness of the test face should be 0.25 μm (10 μin) or finer, as defined by Ra value in ISO 4287.[5] In the latter context, electropolishing or chemical pickling/passivation shall not be used for CRAs. The test specimen shall be fabricated in such way as to avoid overheating and to minimize any incidental cold working of the surface.

5.1.4 A documented cleaning procedure, typically comprising the application of a degreasing agent and rinsing with an appropriate solvent, shall be used to ensure that the test specimens are free of any contaminant, including particulates, oils, greases, or other residual surface films, prior to testing. The surface cleanliness shall be verified using an appropriate method, e.g., ASTM[(3)] F21.[6]

[(3)] ASTM International (ASTM), 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959, www.astm.org.

5.2 Parent Material Specimens

5.2.1 The orientation and position of the specimens with respect to the sample from which they were taken shall be recorded, e.g., longitudinal, near surface, mid-thickness, etc. A typical four-point bend Parent Material specimen is shown in Figure 2(a).

NOTE 1: Specimens may be tested in the As-received condition to avoid grinding induced change(s) in the material properties near surfaces of the specimens.

NOTE 2: Preparation (machining) of specimens from cold-hardened (i.e., cold-worked) stock, e.g., oilfield tubing and casing to ISO 13680[7] groups 2-4, can result in significant changes of strain on the test face. This results from redistribution of residual stresses that may cause unintended under- or over-straining of test face.

5.2.2 Deburring of the edges of the specimen should be undertaken by light manual grinding.

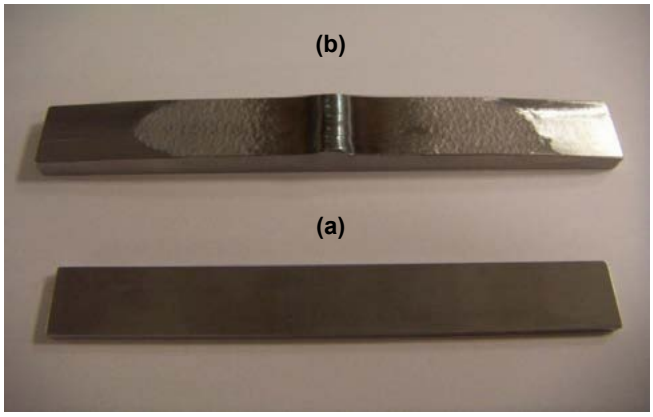

Figure 2: Typical Four-Point Bend Specimens: (a) Parent Material Specimen and (b) As-welded Specimen

5.3 Weldment Specimens

5.3.1 Weldment specimens shall be taken transverse to the weld, with the weld bead at the mid-span of the specimen. A typical four-point bend Weldment specimen is shown in Figure 2(b).

5.3.2 When testing As-welded specimens, the locations in contact with the outer rollers should be machined flat to prevent high localized stress on the rollers due to specimen curvature; otherwise, cracking of the roller (e.g., glass or ceramic rollers) may occur.

NOTE 1: In testing As-welded specimens, there may be inherent lateral curvature of the specimen. The effect of this would induce a higher strain toward the edges of the specimen, although the stress is not much changed. The effect becomes more pronounced for the thicker specimen.

NOTE 2: Dressed weldment specimens may be prepared such that the root/cap bead(s) are intact but dressed flush with the surrounding surface. In this case, care should be taken to avoid damaging and/or modifying the material condition adjacent to the root/cap bead(s).

5.3.3 The variation in thickness of the specimen due to tapering shall be measured and misalignment and curvature (if the weld is machined from a pipe) should be documented by, e.g., photographing with wall thickness measurements.

5.3.4 When preparing Machined Weldment specimens, each test face should be as close as possible to the exposed surface in the field (e.g., retaining as much of the root or cap pass as possible) as there may be hardness and/or microstructural variations through-thickness. There may also be variations in residual stress through the thickness. Therefore, the location of the test face with respect to the original sample surface shall be noted. Specimens shall be cut in a consistent way and the surfaces of all specimens shall be prepared with a consistent finish.

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

5.4 Clad Product Specimens

5.4.1 When testing corrosion resistant alloy specimens in the form of cladding, the carbon steel backing shall be completely removed by machining. This almost inevitably means that thin specimens will be used.

5.4.2 Complete removal of the carbon steel backing shall be verified on each test specimen using an appropriate method, e.g., ASTM A380[8] or a similar standard.

5.4.3 For Weldment specimens, the weld root reinforcement (protrusion) shall be removed. 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 conditions of these regions, in particular the heat tint, may influence the result.

Section 6: Specimen Loading

6.1 The test specimen shall be loaded to the required stress. The stress shall be applied by either setting the deflection measured at the mid-span on the test face (Deflection Method, Paragraph 6.2), and/or by monitoring the applied strain using one or more strain gauges attached directly to the test face (Strain Gauge Method, Paragraph 6.3), in accordance with Table 2. Guidance on strain gauging practices is given in Appendix A (non-mandatory).

Table 2
Specimen Type and Applicable Loading Method

Specimen Type		Applied Stress on Specimen	Load Method(s)
Parent Material	Machined	≤ Elastic limit	Deflection (Paragraph 6.2) or Strain Gauge Method (Paragraph 6.3)
		> Elastic limit	Deflection Method with Calibration [a] (Paragraph 6.2.4) or Strain Gauge Method (Paragraph 6.3)
	As-received	All	Deflection Method with Calibration [a] (Paragraph 6.2.4) or Strain Gauge Method [c] (Paragraph 6.3)
Weldment	Machined	≤ Elastic limit [b]	Deflection Method with Calibration [a] (Paragraph 6.2.6) or Strain Gauge Method (Paragraph 6.3)
		> Elastic limit [b]	Strain Gauge Method (Paragraph 6.3)
	As-welded	All [b]	Strain Gauge Method (Paragraph 6.3)

a: Calibration by the Strain Gauge Method.

b: For welded specimens, the parent metal yield strength is normally used to determine test stresses. For dissimilar joints, the lower parent metal yield strength is normally used. When design stresses are based on the yield strength of a weld zone that is lower than the yield strength of either adjoining parent metals, the yield strength of the weld zone may be used to determine test stresses, as per ANSI/NACE MR0175/ISO 15156-3: B.3.4.

c: Should not be recommended for carbon or low alloy steels in the case that the mill scale removal for strain gauge attachment is not allowed.

6.2 Deflection Method

6.2.1 The objective of the Deflection Method is to achieve a specific value of deflection at the center of the test face of the specimen corresponding to the target longitudinal stress.

6.2.2 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. The displacement monitor shall be positioned at the center of the test face.

NOTE: If a strain gauge on a Parent Material specimen is also positioned at the center of the test face, an adaptor should be attached to the displacement monitor so that it bridges the strain gauge, or the measurement device can be moved toward the edge (width direction) such that it does not contact the strain gauge but retains the location mid-span.

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

6.2.3 For applied stress ≤ the Elastic limit of a Parent Material specimen, Equation (1) may be used to calculate the deflection[9] (y) required to achieve the target longitudinal stress, σ_l. Equation (1) shall not be allowed above the yield strength of the material.

$$y = \frac{(3H^2 - 4A^2)\sigma_l}{12Et} \quad (1)$$

where:
H is the distance between the outer rollers (see Figure 1)
A is the distance between the inner and outer rollers
E is the modulus of elasticity
t is the specimen thickness

NOTE 1: E can be the published or literature value at the test temperature. If literature value at a specific test temperature is not available, E can be accurately identified through the resonance and impact excitation method, as per ASTM E1876.[9] ASTM E111[10] may be used as an alternative method.

NOTE 2: At stresses above the Elastic limit, but below the engineering yield strength, e.g., 0.2% offset or extension under load (EUL), Equation (1) can underestimate the total strain required to load the specimen to the target stress, as shown in Figure 4.

6.2.4 Calibration by Strain Gauge Method (Paragraph 6.3) can be performed on a representative specimen of the test specimens that are made from the same material with the same properties (e.g., chemical composition and yield strength) and identical dimensions. The calibration is to determine the deflection required to obtain the target longitudinal stress on the test face of the calibration specimen equipped with strain gauge. The strain reading is used to indicate if the target applied stress is achieved. The calibrated deflection shall be applied for all the other test specimens to be loaded to the same target applied stress.

6.2.5 For As-received Parent Material specimens loaded to any stress level, or Machined Parent Material specimens loaded to greater than the Elastic limit, the Strain Gauge Method (Paragraph 6.3) is required to load each specimen or otherwise the Deflection Method must be calibrated (Paragraph 6.2.4).

6.2.6 For Machined Weldment specimens loaded to ≤ the Elastic limit of the weaker parent metal, the Deflection Method shall be calibrated with Strain Gauge Method (see Paragraph 6.2.4).

6.2.7 For As-welded specimens loaded to any stress level or Machined Weldment specimens loaded greater than the Elastic limit of weaker parent metal, the Deflection Method shall not be allowed. Each specimen shall be individually loaded with the Strain Gauge Method (see Paragraph 6.3).

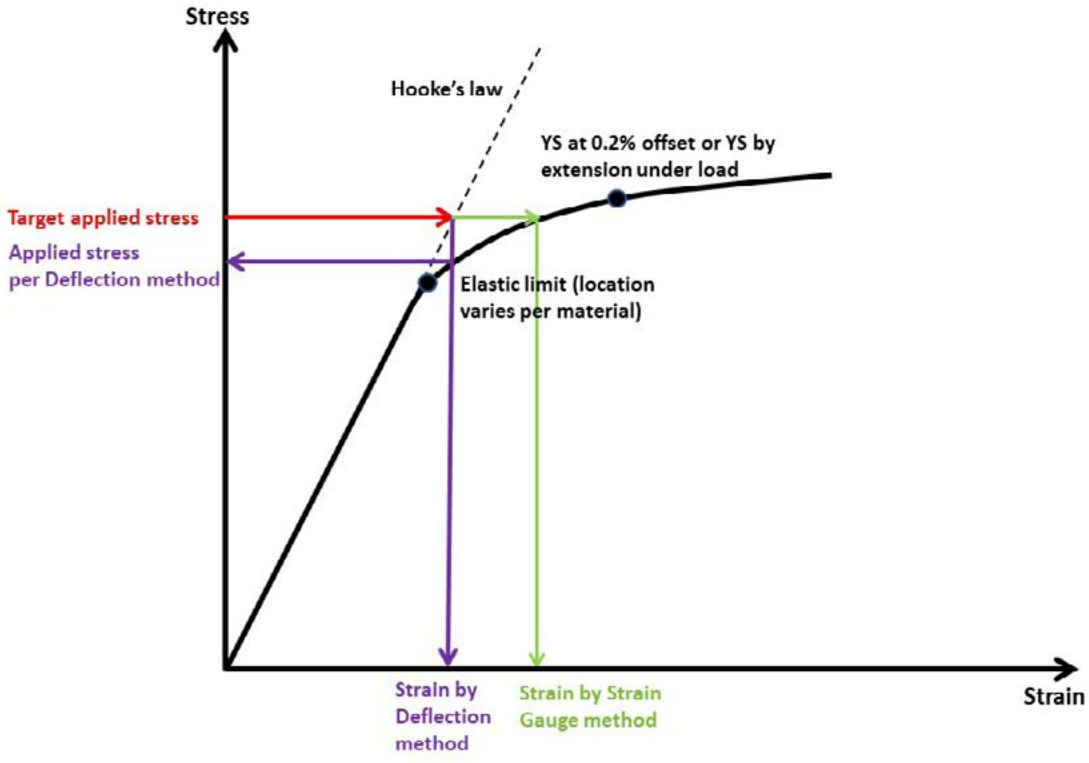

Figure 4: Schematic Stress Strain Curve Showing Elastic Limit, Yield Strength (YS), Potential Strain Underestimate for Target Stress Greater Than Elastic Limit

6.3 Strain Gauge Method

6.3.1 The objective of the Strain Gauge Method is to load the test specimen until the strain measurements indicate the target longitudinal stress is achieved. The Strain Gauge Method can be used for all types of specimens.

NOTE: The Deflection Method with Calibration by the Strain Gauge Method is applicable to Parent Material specimens (see Paragraph 6.2.5) or Machined Weldments loaded to ≤ Elastic limit of the weaker parent metal (see Paragraph 6.2.6).

6.3.2 Attaching strain gauge(s)

For a Parent Material specimen, a strain gauge shall be attached at the center of the test face. The strain gauge should be positioned a minimum of 2x specimen thickness away from the (inner) roller centerline. Near the inner roller there is an additional confining stress due to the friction on specimen by the roller.

For Weldment specimens, strain gauges shall be attached at mid-width and near mid-span of the test face of the specimen symmetrically on each side of the weld. Strain gauges should be located on the parent metal as close as possible to the weld, but outside the HAZ and at least a distance of 2x specimen thickness from the nearest of the inner rollers, unless prevented by the dimension of large welds (e.g., welds with weld cap).

Machined Weldment specimens stressed ≥ Elastic limit of the weaker parent metal or As-welded specimens shall be individually strain gauged.

6.3.3 Loading Specimens stressed ≤ the Elastic limit

For Parent Material specimens, the application of the target longitudinal stress is controlled either via formula derived from Hooke's Law or from stress-strain curve obtained from uniaxial tensile testing (see Paragraph 6.3.4).

When biaxial strain gauges are used to stress Parent Material ≤ the Elastic limit or Machined Weldment specimen ≤ the Elastic limit, the longitudinal stress is calculated by using Equation (2) with the measured strain gauge data ε_t and ε_l:

$$\sigma_l = \frac{E}{1 - v^2} (\varepsilon_l + V\varepsilon_t) \quad (2)$$

where:
v is Poisson's ratio
ε_l l ongitudinal strain
ε_t is transverse strain

NOTE 1: Uniaxial strain gauges are often sufficient for CS, LAS and Martensitic Stainless Steel (MSS) Parent Materials. Biaxial strain gauges are recommended for all types of Weldments, and anisotropic materials, e.g., Duplex and Austenitic Stainless Steels (DSS and ASS).

When uniaxial strain gauge is used for a Parent Material or Machined Weldment specimen, the longitudinal stress is calculated by using Equation (3) with the measured strain gauge data :

$$\sigma_l = E * \varepsilon_l \quad (3)$$

When using Equation (2) or (3) for stress application, the test specimen shall be loaded until the target longitudinal stress σ_l is reached.

NOTE 2: Equation (2) reduces to Equation (3) for any case in which a uniaxial state of stress is achieved, i.e., Equation (4) is true. Confirmation of Equation (4) can be obtained for materials in a uniaxial state of stress measured with biaxial strain gauge(s).

$$\varepsilon_t = \nu * (-\varepsilon_l) \quad (4)$$

NOTE 3: In the presence of a biaxial stress state, the use of a single-axis strain gauge to set the total longitudinal strain will result in the conservative loading (over-stressing) of the test specimen. Local buckling of weldment test specimens has been reported in some cases.

6.3.4 Loading Specimens stressed > Elastic limit

For all specimens including Parent Material and Weldment specimens, the specimens shall be loaded until the total measured strain (elastic and plastic) matches the desired loading condition (target strain). Correlating the strain gauge reading to the desired loading condition (stress, plastic strain, etc.) shall be performed using uniaxial tensile stress-strain data[7] at the test temperature.

NOTE 1: Uniaxial stress-strain data is recorded in terms of engineering stress and strain. Strain gauges also read engineering strain.

NOTE 2: The uniaxial stress-strain data should be based on at least two separate tensile tests (see ISO 6892,[11] ASTM E8[12] or ASTM E21[13]) using specimens taken adjacent to, and in the same orientation as, the Four-Point Bend test specimens. The target strain for loading the specimen should be averaged from multiple stress-strain curves. Strain values taken directly from tensile tests may not be accurate.

6.3.5 Special loading requirements for Weldment specimens

For Weldment specimens with the same parent metal on each side of the weld, the applied strain shall be determined when one of the strain gauges on either side of the weld first registers the required strain in the parent metal.

For the Weldment with dissimilar parent metals, the applied strain shall be determined when the weaker parent metal with the lower YS reaches the desired loading condition.

6.3.6 Attachment and subsequent removal of the gauges shall be undertaken in such a way so as to minimize changes in the surface state. Care shall be taken to minimize the area affected. Cleaning in accordance with Paragraph 5.1.4 may be sufficient.

Section 7: Test Environment

7.1 General

7.1.1 For testing to be performed in a test environment simulating a specific service application, or under standard conditions, such as NACE TM0177 Solutions A, B, C or D,[14] preparation of the test environment shall follow the practices outlined by NACE TM0177.

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

7.1.2 The test vessel material shall not lead to contamination of the test environment under the specified test conditions.

7.1.3 A documented cleaning procedure shall be used to ensure that the testing equipment, including the test vessel and associated accessories in contact with the test environment, are free of all contaminants, prior to testing.

7.1.4 When testing specimens made of carbon and low alloy steels, the test vessel shall be sized to maintain a test solution volume of at least 20 mL/cm^2 of test specimen surface area.[14]

7.1.5 Methods of deaeration and transfer of test solution to the test vessel shall be used that result in a sufficiently de-aerated test solution. The oxygen concentration in the test solution shall be maintained below 10 ppb when testing stainless steels and corrosion resistant alloys or when testing carbon and low-alloy steels with actual strength level (AYS) ≥ 80 ksi (552 MPa). The oxygen concentration in the test solution shall be maintained below 50 ppb when testing carbon or low-alloy steels with AYS <80 ksi (552 MPa). 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.

7.1.6 Monitoring of the oxygen concentration in every test is not mandatory. However, a separate test shall be conducted using the same apparatus and procedure, to demonstrate that the methodology and procedure can achieve the required level of oxygen. Evidence showing achievement of the required oxygen level shall be documented.

7.1.7 The test temperature shall be maintained and monitored within ± 3 °C of the target value.

7.2 pH Adjustment and Control

7.2.1 The solution pH may be fixed either by the specified aqueous solution 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. The guidance of NACE TM0177 for pH adjustment and control shall apply.

7.2.2 The test solution pH shall be measured at the start of the test and at the end of the test. If adjustments of the pH during test are required additional pH values shall be measured and reported.

NOTE: Where testing is performed in NACE TM0177 Environments A or B,[14] the respective solution pH requirements shall apply.

Section 8: Procedure for Four-Point Bend Testing

8.1 The required deflection or strain for loading the specimen shall be determined, as described in Section 6.

8.2 The specimen shall be placed in the loading jig and loaded to the required deflection or strain as prescribed in Section 6. The target deflection/the actual deflection or the actual resultant strain shall be recorded. In all cases, the extent of initial stress relaxation together with description of the adjustments shall be recorded.

NOTE: Stress relaxation should be avoided, and methodology to mitigate stress relaxation should be documented. Known contributors to stress relaxation may include specimen redistribution of stress during loading, jig design, and/or elevated temperature relaxation of the specimen stress and/or jig response, etc.

Stress relaxation upon loading may be observed by reductions or redistributions in strain. As such, after loading to the target strain, the strain gauge values should be monitored to determine the level of constancy. The criterion for evaluating strain constancy is strain deviation within ± 2% of target as observed over the course of 30 minutes. If the strain constancy criterion is not met, the deflection should be adjusted so as to attain the target strain and strain constancy should be re-evaluated.

8.3 Before testing, the test specimen and the loading jig shall be cleaned in accordance with Paragraphs 5.1.4 and 7.1.3, respectively.

8.4 If the test is going to be performed at an elevated temperature (typically above 50 °C/120 °F), Section 7 of TM0177 fully applies, and the procedure described in the following Paragraphs 8.5-8.13 shall be adapted accordingly.[14]

8.5 The specimen (in its loading jig) shall be placed in the test vessel, then the lid shall be sealed and checked for leaks.

NOTE: Prior to immersion, the test specimen should be stored in such a way to minimize any impact of the atmosphere on subsequent performance.

8.6 The test solution shall be added to the test vessel in such a way as to meet the requirements of Paragraph 7.1.5 on residual oxygen content. Paragraphs 8.7-8.10 provide an often suitable, though not exclusive, methodology for mitigating O_2 ingress.

8.7 The test solution should be placed in a separate reservoir and deaerated by purging with a suitable low oxygen purging gas (see Paragraphs 7.1.5 and 7.1.6). 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 a number of parameters, including the size of the gas bubbles, the period in contact with the test solution, and the flow rate.[14]

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. Upon charging with the test gas especially containing CO_2, equilibrium will be restored when CO_2 re-dissolves in the solution (can be confirmed by pH measurement after testing).

8.8 The test vessel and connecting tubes shall be deaerated prior to transfer of the solution from the reservoir to ensure no oxygen contamination.

8.9 The solution may be transferred into the test vessel using an appropriate method, e.g., the pressure of the purging gas, gravity, or pumping.

8.10 The solution shall be saturated with the test gas using a flow rate and bubble size appropriate to attain 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. Alternative charging rates may be adopted for larger vessels, for example, evidence showing attainment of saturation shall be documented. The gas saturation shall be achieved by continuous or periodic replenishment as described in NACE 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 should be provided.

NOTE: For testing of carbon or low alloy steels with a sealed-in system with no replenishment of test gas, depletion of H_2S may occur during the test. The concentration of dissolved H_2S can be measured during the test and/or at the end of the test.

8.11 Upon attainment of the specified test conditions, the exposure period shall begin. The test conditions shall be maintained throughout the specified exposure period.

8.12 At the end of the exposure period, dissolved H_2S shall be removed. This may be achieved by sparging the test solution with nitrogen gas.

8.13 The post-test specimen before being removed from the jig shall be appropriately cleaned and dried, e.g., rinsed with water and acetone, and blown dry using nitrogen gas. Further cleaning and drying of the post-test specimen may be performed after removal from the jig.

8.14 The post-test specimen shall be photographed as-tested (and/or after removal of corrosion product if performed).

NOTE 1: An inhibited HCl solution may be used to remove corrosion product for low alloy or carbon steels (see ASTM G1).[15]

NOTE 2: H_2S may be released when the corrosion products are being dissolved with acid solution (see Appendix C for safety considerations in handling H_2S toxicity).

Section 9: Specimen Evaluation

9.1 For unfractured (nonbroken) test specimens, any evidence of corrosion attack, including surface breaking cracking, sub-surface cracking, and localized corrosion shall be evaluated.

9.1.1 Visual examination with 10X magnification shall be performed for the entire surface of the specimens.

9.1.2 The following methods enable increasingly more detailed examinations of specimens for cracking and localized corrosion. For steels, these methods may be used whenever suspicious feature (e.g., localized corrosion and/or potentially sub-surface cracking) is observed in Paragraph 9.1.1. For CRA materials, the following methods shall be performed even without any suspicious feature.

(i) Non-destructive examinations, such as Magnetic Particle Inspection (MPI),[16] liquid/dye penetrant testing,[17] or Ultrasonic Testing (UT)[18] for the entire surface of the specimens.

(ii) Metallurgical examinations of the evaluation section of the test face in the case where suspicious feature(s) is identified in Paragraph 9.1.2 (i). The examinations shall be performed for each type of suspicious features identified. The specimens should be prepared with longitudinal sectioning followed by metallographic preparation of the cut faces and through-thickness examinations in the unetched condition up to 100X magnification. The type, extent, and location of any cracking shall be confirmed in the etched condition.

(iii) Metallurgical examinations for the evaluation section of the test face of the specimens, in the case where no surface cracking or any suspicious feature is identified in Paragraphs 9.1.1 and 9.1.2 (i). The specimens can be prepared with longitudinal sectioning (typically at mid-width, or at 1/3 and 2/3 of width for thicker wall specimens) followed by metallographic preparation of the cut faces and through-thickness examinations in the unetched condition at 100X magnification or greater (e.g., 200-500X can detect micro-cracks or micro-features). The type, extent, and location of any cracking shall be confirmed in the etched condition.

9.2 All cracking identified shall be documented photographically and reported, including the type, extent, and location of the cracking. All fractured specimens shall be reported with photographs.

NOTE 1: 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.

NOTE 2: Although this test method is intended for the determination of SCC or SSC resistance, other types of cracking, e.g., hydrogen-induced cracking (HIC), stepwise cracking (SWC), stress-oriented hydrogen-induced cracking (SOHIC), and pitting corrosion may also be observed during specimen evaluation.

9.3 An unstressed and unexposed reference specimen may be evaluated as reference for any evidence of cracking in the test specimens identified in Paragraph 9.1.

NOTE: Cracking or crack-like flaws may be produced during material processing/welding and could be confused with cracking resulting from environmental testing.

9.4 The visually and/or metallographically observed corrosion pits or other notable features shall be recorded photographically and reported.

NOTE: An assessment of the severity of any corrosion pitting can be made by determining the maximum pit depth and evaluating the shape of pits (see ISO 11463).[19]

Section 10: Test Report

The test report shall conform to all reporting requirements in the material manufacturing specification or material requirements document specified. As a minimum, the test report shall include all relevant information as follows:

a) full description of the test material, e.g., source (or origin), heat number, heat treatment lot, mechanical properties, composition and structural condition, type of product, welding parameters, etc.;
b) the target stress and applied deflection and/or strain;
c) information of the specimens, including specimen type (see Table 2), location from which the specimens were removed, position of specimens with respect to the root or cap face of the original weld (e.g., 3 mm from root face), orientation of the specimens, dimensions (including any non-uniformity of thickness), surface preparation;
e) details of the loading procedure adopted (i.e., strain gauge, deflection measurement, use of calibration specimens, etc.), including all relevant calculations, values of elastic modulus and other material parameters employed, derivation of target strain gauge and/or deflection values, and the actual loaded values attained for each individual test specimen;
f) environmental parameters:
    i. Test solution composition and/or information;
    ii. initial pH, pH before and after saturation by the test gas if measured; final pH and any pH adjustment made during test;
    iii. composition for test gas and de-aeration gas with impurity;
    iv. If measured, dissolved O_2 of de-aerated test solution;
    v. if measured, dissolved H_2S after gas saturation at the end of test and during test;
    vi. test temperature;
    vii. exposure time.
g) method(s) used for evaluating cracking and examination magnification(s);
h) report the location, type, and extent of any cracking and localized corrosion identified in Section 9;
i) Photograph of post-test specimen as-tested and/or after removal of corrosion product if performed.

References

1. ANSI/NACE MR0175/ISO 15156-1 (latest revision), “Petroleum and Natural Gas Industries — Materials for Use in H_2S-containing Environments in Oil and Gas Production — Part 1: General Principles for Selection of Cracking-resistant Materials” (Houston, TX: AMPP).

2. ANSI/NACE MR0175/ISO 15156-2 (latest revision), “Petroleum and Natural Gas Industries — Materials for Use in H_2S-containing Environments in Oil and Gas Production —- Part 2: Cracking-resistant Carbon and Low Alloy Steels, and the Use of Cast Irons” (Houston, TX: AMPP).

3. ANSI/NACE MR0175/ISO 15156-3 (latest revision), “Petroleum and Natural Gas Industries — Materials for Use in H_2S-containing Environments in Oil and Gas Production — Part 3: Cracking-resistant CRAs (Corrosion-resistant Alloys) and Other Alloys” (Houston, TX: AMPP).

4. ASTM C1161 (latest revision), “Standard Test Method of Advanced Ceramics at Ambient Temperature” (West Conshohocken, PA: ASTM).

5. ISO 4287 (1997 version), “Geometrical Production Specification (GPS) – Surface Texture: Profile method – Terms, definition and surface texture parameters” (Geneva, Switzerland: ISO).

6. ASTM F21 (latest revision), “Standard Test Method for Hydrophobic Surface Films by the Atomizer Test” (West Conshohocken, PA: ASTM).

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9. ASTM E1876 (latest revision), “Standard Test Method for Dynamic Young’s Modulus, Shear Modulus, and Poisson’s Ratio by Impulse Excitation of Vibration” (West Conshohocken, PA: ASTM).

10. ASTM E111 (latest revision), “ Standard Test Method for Young’s Modulus, Tangent Modulus, and Chord Modulus” (West Conshohocken, PA: ASTM).

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

12. ASTM E8/EM (latest revision), “Standard Test Methods for Tensile Testing of Metallic Materials” (West Conshohocken, PA: ASTM).

13. ASTM E21 (latest revision), “Standard Test Methods for Elevated Temperature Tension Tests of Metallic Materials” (West Conshohocken, PA: ASTM).

14. NACE TM0177 (latest revision), “Standard Test for Laboratory Testing of Metals for Resistance to Sulfide Stress Cracking and Stress Corrosion Cracking in H_2S Environments” (Houston, TX: AMPP).

15. ASTM G1 (latest revision), “Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens” (West Conshohocken, PA: ASTM).

16. ASTM E3024/E3024M (latest revision), “Standard Practice for Magnetic Particle Testing for General Industry” (West Conshohocken, PA: ASTM).

17. ASTM E1417/E1417M (latest revision), “Standard Practice for Liquid Penetrant Testing” (West Conshohocken, PA: ASTM).

18. NACE TM0284 (latest revision), “Evaluation of Pipeline and Pressure Vessel Steels for Resistance to Hydrogen-Induced Cracking” (Houston, TX: AMPP).

19. ISO 11463 (latest revision), “Corrosion of metals and alloys - Evaluation of pitting corrosion” (Geneva, Switzerland: ISO).

20. British Society for Strain Measurement (BSSM)[4] CP1 (latest revision), “Code of Practice for the Installation of Electrical Resistance Strain Gauges” (Uffington, Faringdon, U.K.: BSSM).

21. Chemical Safety Data Sheet SD-36 (Washington, DC: Manufacturing Chemists Association, 1950).

22. N. Irving Sax, *Dangerous Properties of Industrial Materials* (New York, NY: Reinhold Book Corp., 1984).

[4] British Society for Strain Measurement (BSSM), 2 Craven Common, Uffington, Faringdon SN7 7RN, U.K., www.bssm.org.

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[5] European Federation of Corrosion (EFC), 1 Carlton House Terrace, London SW1Y 5DB, U.K., efcweb.org.
[6] The Institute of Materials, Minerals and Mining, 297 Euston Road, London NW1 3AQ, U.K., www.iom3.org.

Appendix A
Procedure for Strain Gauging Installation
(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.

A useful reference for strain gauge installation is the British Society for Strain Measurement (BSSM) code of practice CP1.[20]

- Lightly abrade the surface using 400 grade silicon carbide paper

  NOTE: For As-received parent pipe specimens from carbon or low alloy steels, the calibrated Deflection Method is recommended to avoid artificially remove mill scale on the test specimens. For As-welded specimens from carbon or low alloy steels, abrasion can be done only on the parent pipe. For As-received parent pipe or As-welded specimens from stainless steels and CRAs, the test face is smooth enough without further abrasion.
- 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 specimen 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 logbook or on worksheets

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):

$$E = \sigma / \varepsilon \quad \text{(B1)}$$

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

$$\sigma = \frac{3d_1 W}{wt^2} \quad \text{(B2)}$$

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
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[7] maximum allowable concentration of H_2S in the air for an eight-hour workday is 20 mg/L, well above the level detectable by smell. 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[21] and from *Dangerous Properties of Industrial Materials*.[22]

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.

[7] Occupational Safety and Health Administration (OSHA), U.S. Department of Labor, 200 Constitution Ave. NW, Washington, DC 20210, www.osha.gov.