# INTERNATIONAL STANDARD

# ISO 16540

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# Corrosion of metals and alloys — Methodology for determining the resistance of metals to stress corrosion cracking using the four-point bend method

*Corrosion des métaux et alliages — Méthodologie de détermination de la résistance des métaux à la fissuration par corrosion sous contrainte au moyen de la méthode de flexion quatre points*

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# Foreword

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The committee responsible for this document is ISO/TC 156, *Corrosion of metals and alloys*.

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# Introduction

This International Standard has been prepared as a sub-set of ISO 7539 which consists of the following parts, under the general title *Corrosion of metals and alloys — Stress corrosion testing*:

— *Part 1: General guidance on testing procedures*

— *Part 2: Preparation and use of bent-beam specimens*

— *Part 3: Preparation and use of U-bend specimens*

— *Part 4: Preparation and use of uniaxially loaded tension specimens*

— *Part 5: Preparation and use of C-ring specimens*

— *Part 6: Preparation and use of precracked specimens for tests under constant load or constant displacement*

— *Part 7: Method for slow strain rate testing*

— *Part 8: Preparation and use of specimens to evaluate weldments*

— *Part 9: Preparation and use of pre-cracked specimens for tests under rising load or rising displacement*

— *Part 10: Reverse U-bend method*

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

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# Corrosion of metals and alloys — Methodology for determining the resistance of metals to stress corrosion cracking using the four-point bend method

## 1 Scope

This International Standard 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. The method gives guidance on testing of both parent plate and welds and includes procedures for metals that have no distinct yield point in their stress-strain behaviour as well as metals with a distinct yield point. The emphasis in this International Standard is on the generic methodology of the four-point bend test. Service application will be varied and the relevant industry standard is to be consulted where appropriate.

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## 2 Normative references

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

ISO 8407, *Corrosion of metals and alloys — Removal of corrosion products from corrosion test specimens*

## 3 Terms and definitions

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

**3.1**
**corrosion resistant alloy**
**CRA**
alloy designed to be resistant to general and localized corrosion in environments that are corrosive to carbon steel

**3.2**
**heat affected zone**
**HAZ**
portion of the base metal that is not melted during brazing, cutting, or welding, but whose microstructure and properties are altered by the heat of these processes

**3.3**
**soft zone cracking**
**SZC**
form of sulphide stress cracking that may occur when a steel contains a local "soft zone" of low yield strength material and is exposed under stress to environments containing $H_2S$

Note 1 to entry: Under service loads, soft zones may yield and accumulate plastic strain locally increasing the susceptibility to cracking of an otherwise cracking resistant material. Such soft zones are typically associated with welds in carbon steels.

## 4 Principle

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

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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 decrease. Hence, complete fracture might 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.

## 5 Loading jig design

**5.1** 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.

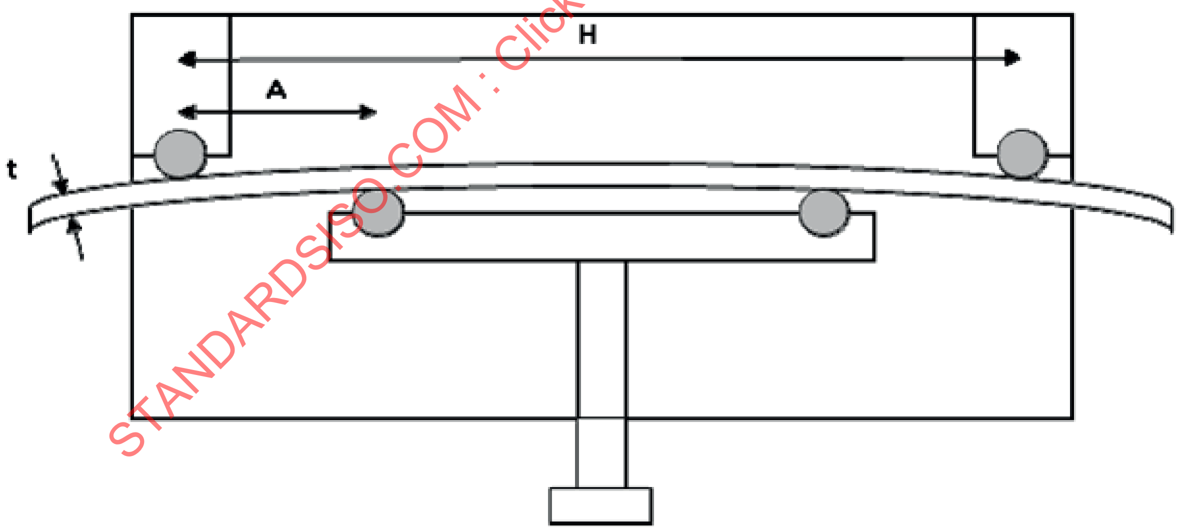
**5.2** Specimens of thickness up to 5 mm present few problems for parent material specimens as they can be easily accommodated in test vessels of modest size with typical dimensions for the loading jig of the following:

— spacing between inner rollers: 50 mm-60 mm;

— spacing between outer rollers: 100 mm-130 mm;

— roller diameter: 6 mm-10 mm.

**5.3** 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.




**Key**

t specimen thickness

A distance between the inner and outer supports

H distance between the outer supports

**Figure 1 — Typical four-point bend loading jig design**

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**5.4** 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.

**5.5** Friction between the rollers (bearing cylinders) and the specimen should be minimised to ensure that frictional constraint does not impact 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 can be further reduced if they are free to rotate while loading the test specimen. 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 test results. Nevertheless, an increase in friction will increase the stress and strain on the tensile surface locally at the inner loading pins and can enhance the likelihood of cracks forming in the specimen at those locations (see Clause 11). The extent of overstraining for a particular loading jig can be assessed by strain gauging in that region for a typical test condition.

**5.6** 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 minimized to avoid impacting on the test results. This can 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 undertaken, 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.

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## 6 Specimen preparation

### 6.1 General

**6.1.1** 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 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).

**6.1.2** 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.

**6.1.3** Specimen preparation techniques which 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 can also be considered, but only where this does not introduce changes in the material microstructure/microchemistry.

### 6.2 Parent material specimens

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

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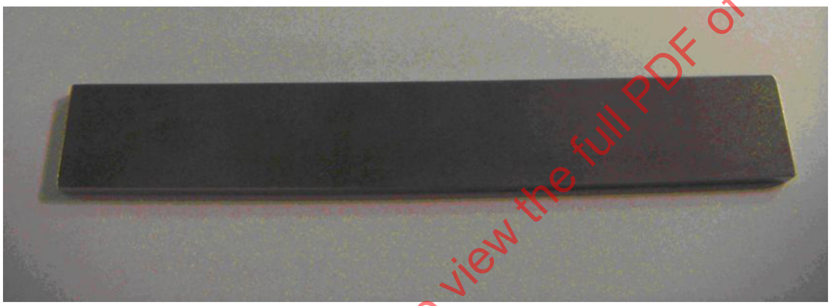
**6.2.2** A typical four-point bend parent material specimen is shown in Figure 2 a).

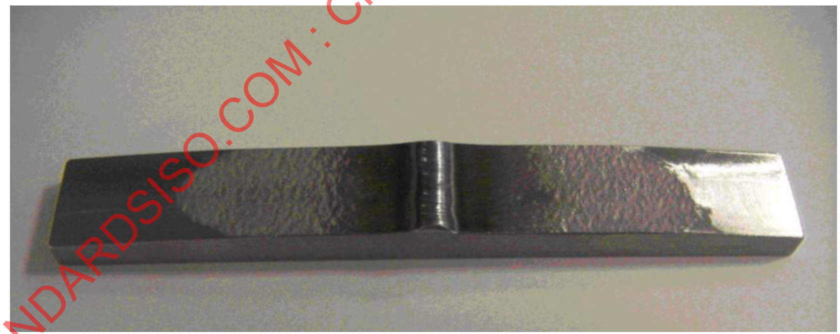
**6.2.3** The specimen width shall be 1,5 to 5 times the thickness of the specimen. Any deviation from this requirement, e.g. for very thick C-steel sections, requires demonstration that out-of-plane bending is not significant.

**6.2.4** The specimen can 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 an Ra value, ≤0,25 µm, for any non-welded specimen. 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. The effectiveness of all cleaning procedures adopted in this International Standard shall be demonstrated, for example, using an atomizer test.[9]

**6.2.5** Deburring of the edges of the specimen can be undertaken by light manual grinding.



**a) parent material specimen**



**b) as-welded specimen**

**Figure 2 — Typical four-point bend specimens**

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## 6.3 Welded specimens

**6.3.1** Unless specified otherwise, welded specimens shall be taken transverse to the weld where feasible with the weld bead at the centre of the specimen.

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

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**6.3.3** 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 can often result in 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.

**6.3.4** 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 ceramic supports due to specimen curvature. Otherwise, cracking of the roller can occur.

**6.3.5** For both fully machined and as-welded specimens, the specimen width shall be 1,5 to 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.

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

**6.3.7** When testing welds under fully machined conditions, the surface under tension should be as close as possible to the original surface as there might be hardness and microstructural variations through-thickness. In particular, the root pass (or final pass if testing relates to the weld cap surface) 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 can also be variations in residual stress through the thickness. Accordingly, the location of the specimen surface in tension with respect to the original surface shall be noted and specimens cut in a consistent way. The surface shall be prepared in accordance with 6.2.4 to a consistent repeatable finish as agreed with end-user, but usually with an 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 6.2.4.

**6.3.8** Deburring of the edges of the specimen can be undertaken by light manual grinding.

### 6.4 Clad product specimens

**6.4.1** When testing corrosion resistant alloy specimens from clad pipe or pressure vessel wall, the carbon steel backing shall be completely removed by machining. This inevitably means that thin specimens need to be used.

NOTE The efficacy of removal of the carbon steel backing can be checked by using the copper sulfate test, for example.[8]

**6.4.2** 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 HAZ/parent regions since the surface condition of these regions, in particular the heat tint, can influence the result.

## 7 Strain gauging

**7.1** Strain gauging is required when the loading of the specimen is such that it could induce plastic deformation. Guidance on strain gauging is given in Annex B.

**7.2** 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 centre of the face in tension.

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**7.3** For testing of welded specimens, strain gauges shall be attached to the parent material in the centre 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 heat affected zone (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.

**7.4** 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 in 6.2.4. Care shall be taken to minimize the area affected.

# 8 Loading

## 8.1 Strain level

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

## 8.2 Setting the total strain value

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

**8.2.2** The required deflection shall be measured at the centre 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.

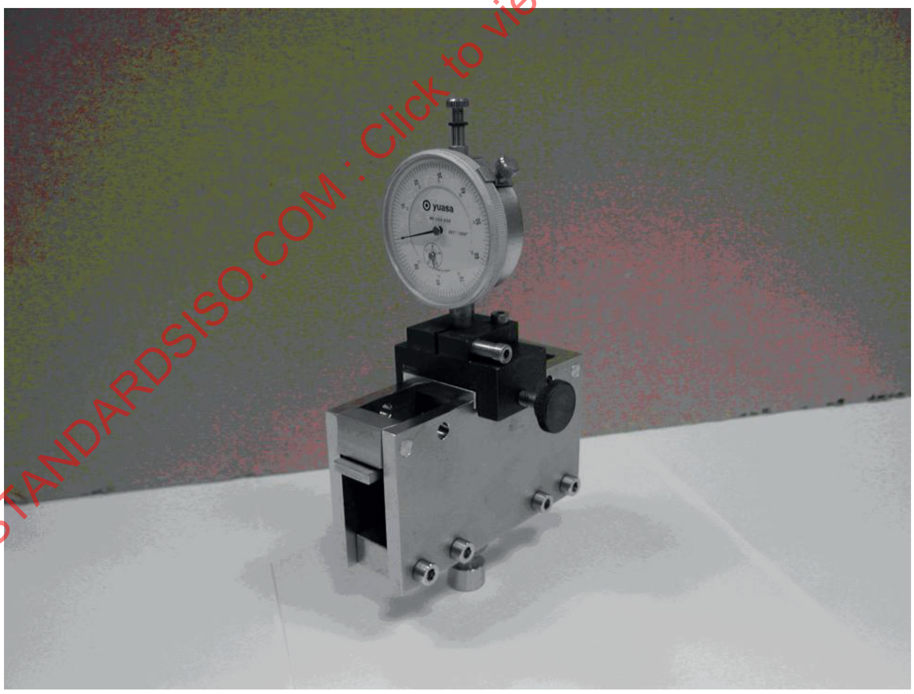

**Figure 3 — Loading jig with dial gauge attached for measurement of deflection**

**8.2.3** For applied stresses below the elastic limit, Formula (1) should be used to set the deflection, *y*.

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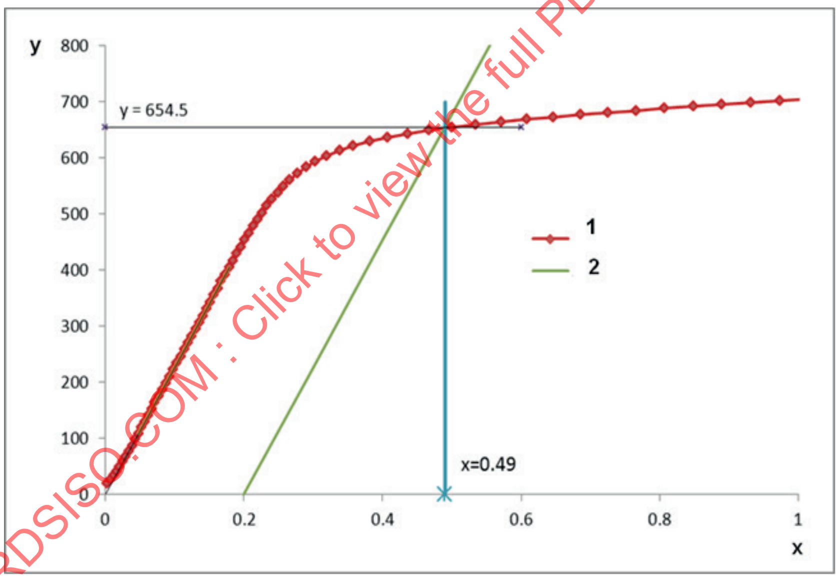
$$y = \frac{\left(3H^2 - 4A^2\right)\sigma}{12Et} \quad (1)$$

where $\sigma$ 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, Formula (1) is then valid up to the yield point (the lower yield point in this case).

**8.2.4** 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. The uniaxial data shall be based on three separate tensile tests using specimens prepared from the same heat treatment batch close to the location of source material and with the same orientation from which the four-point bend test specimens are obtained. The tensile specimens shall be the form of parent material fully machined and ground to the surface finish specified in 6.2.4.

**8.2.5** 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).




**Key**

*y* uniaxial stress (MPa)

*x* uniaxial strain

1 actual experimental data

2 0,2 % offset

**Figure 4 — Typical example of stress-strain data for a corrosion resistant alloy showing determination of total strain to be applied to achieve 0,2 % plastic strain in four-point bend testing**

**8.2.6** When testing parent material, the deflection to be applied to the four point bend specimen shall be determined by undertaking a calibration test in which the deflection is measured as a function of applied load until the required total strain is achieved. For calibration specimens, the deflection shall be measured using a suitable displacement monitor (6.2.2). Since the strain gauge on the calibration

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specimen is also positioned at the centre of the face in tension, an adaptor shall be attached to the displacement monitor so that it bridges the strain gauge.

**8.2.7** 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. Since 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 total strain derived from the uniaxial tensile test.

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

**8.2.9** Any specimen strained beyond 1 % of the intended level shall be discarded or tested in the overstrained condition.

## 8.3 Testing at elevated temperature

**8.3.1** The mechanical properties of the material will change with temperature, the proof stress usually decreasing. This decrease is more marked for austenitic and 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.

**8.3.2** For testing at elevated temperature, the total strain corresponding to 0,2 % plastic strain at the specified test temperature shall be determined from the stress-strain curve derived from a uniaxial tensile test on a reference specimen of the parent material at the test temperature (see Annex B). The test specimen shall then be loaded at room temperature 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.

**8.3.3** 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.

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# 9 Test environment

## 9.1 General

**9.1.1** The test environment in terms of the solution composition and test gases should reflect the intended application. General guidance is given in ISO 7539-1.

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

**9.1.3** Tests at atmospheric pressure can be carried out in a glass vessel. Tests at elevated pressure shall be carried out in an autoclave.

**9.1.4** For tests in deaerated solution using glass vessels, a nitrogen cabinet can be used as sealing of the lid might not be as effective as that on an autoclave. The oxygen concentration in the test solution shall be maintained below 10 parts per billion by mass. In some applications, a slightly higher oxygen concentration can be tolerated provided it is demonstrated that there is no effect on the test result.

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**9.1.5** Validation of the methodology for attaining the required oxygen concentration shall be demonstrated in a separate test using the same apparatus and procedure, but with an oxygen concentration monitor.

**9.1.6** The test temperature shall be maintained within ±3 °C, unless otherwise specified.

**9.1.7** When calculating the total pressure in tests at elevated temperature, 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 documented in detail.

**9.1.8** All chemicals used shall be high purity grades.

## 10 Procedure for four-point bend testing

**10.1** Determine the required deflection for the specimen as described in Clause 8.

**10.2** 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.

**10.3** Place the specimen in the loading jig and load the specimen to the required deflection or strain.

**10.4** Check for any strain relaxation as follows. 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.

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

**10.6** For tests in aerated solution, it is sufficient to simply add the test solution to the test vessel.

**10.7** For tests in deaerated solution, the procedure adopted shall avoid transient exposure to oxygen in solution. In this case, place the test solution in a separate reservoir and deaerate by purging with nitrogen. A period of 1 h per litre of test solution is usually sufficient at typical flow rates (~0,1 L/min $N_2$).

**10.8** 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.

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.

**10.9** Pump the solution into the test vessel using the pressure of the purging gas. Saturate the solution with the test gas. The gas concentration in solution shall be maintained at the required level during the test. This can be achieved by continuous or periodic replenishment.

**10.10** 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.

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

**10.12** Subsequently remove the specimens, clean with water, and dry with acetone.

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

**10.14** If necessary, remove any corrosion product following the procedures in ISO 8407.

## 11 Failure appraisal

### 11.1 Carbon steel

**11.1.1** Test specimens shall be evaluated for any evidence of cracking including surface breaking cracks, sub-surface/surface breaking stress oriented hydrogen-induced cracking (SOHIC), and soft zone cracking (SZC).

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

a) initial visual examination at ×10 magnification;

b) non-destructive assessment of the presence of cracks using, for example, magnetic particle inspection (MPI) or liquid penetrant for surface cracks on the stressed test face or ultrasonic testing;

c) sectioning of the specimens at any suspicious features noted in steps a) and b);

d) 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 100× magnification of cut faces. The size and location of any cracks shall be confirmed in the etched condition.

**11.1.3** 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 loading pins because of an elevated local stress and strain compared to that in the stressed region between the rollers.

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

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).

**11.1.5** An unstressed reference specimen shall be evaluated for any evidence of cracking as per 11.1.2.

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.

### 11.2 Corrosion resistant alloys

**11.2.1** Examine specimens using low powered microscopy to at least ×10 magnification. Photographic evidence of any cracking shall be recorded.

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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.

**11.2.2** Visual/low powered microscopic examination can be complemented by dye penetrant examination (DPE) or fluorescent dye penetrant examination (FDPE).

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

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).

**11.2.4** Where no surface cracks are visible, undertake longitudinal sectioning at two locations (typically at 1/3 and 2/3 through-thickness) and conduct metallographic examination at up to ×100 magnification.

**11.2.5** An unstressed reference specimen shall be evaluated for any evidence of cracking as per 11.2.2 and 11.2.3.

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

## 12 Test report

As a minimum, the test report shall include a reference to this International Standard, i.e. ISO 16540 and 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 any 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, photographic evidence of pitting (if any), maximum pit depth when measured.

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# Annex A
(informative)

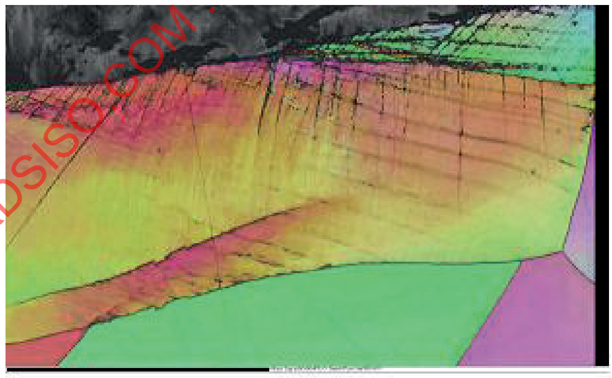
# Surface preparation (see also ISO 7539-1)

## A.1 General

The role of laboratory testing is twofold: to generate repeatable and reproducible data and to provide data relevant to the application. In the former case, the usual specification in standards is average surface roughness, Ra, less than a certain maximum which can range from 0,2 µm to 0,8 µm, while for service simulation, the surface condition will aim to reflect the specific application which will be highly variable depending on the material, nature of the component/product, and aggressivity of the environment. There is a lack of general awareness of the impact of surface preparation on near-surface properties including not only surface roughness, but physical defects, residual stress, near-surface microstructure (including phase transformation due to cold work) and mechanical properties, and the nature of the oxide (transformed by welding perhaps).

## A.2 Surface deformation

A fundamental objective in surface preparation is that the process adopted should be undertaken in a step-wise manner so that any prior history in terms of near surface properties should be removed at each stage of grinding or machining. Otherwise, residual sub-surface deformation might be retained in the specimen, which otherwise might appear to meet the requirements of the specified Ra value. Metallographic examination in section using electron back-scattered diffraction (EBSD) should be used on a reference specimen to assess the degree of sub-surface deformation and conformity to the required procedure. Figure A.1 illustrates a “dressed” surface[11] with an Ra value of 0,2 with very evident slip lines showing that progressive removal of prior deformation history had not been sufficient.



NOTE The scale bar on the bottom of this figure is 50 µm.

**Figure A.1 — EBSD image for transverse dressed UNS S30403**

Figurer A.2 shows an EBSD orientation map of a ground specimen with an Ra value of 0,6. In this case, the slip lines are not evident, but a nanocrystalline layer is apparent that was removed effectively in the case of the dressing process. This is not related to a smaller value for Ra since this nanocrystalline layer exists for a ground surface with Ra of 0,2, though the finer the surface finish the less depth of surface deformation.

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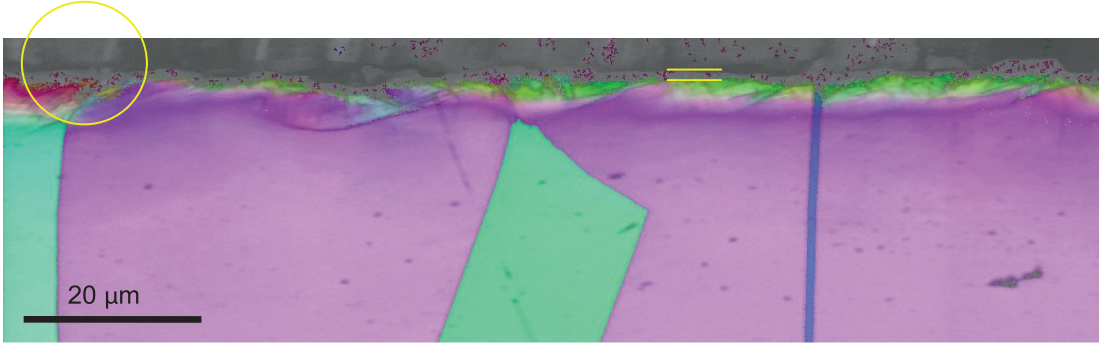


NOTE Nanocrystalline layer between two yellow horizontal lines approximately 2 µm thick (small purple dots within and above this region largely incorrectly indexed pixels) and deformation of a grain boundary (circled).

**Figure A.2 — EBSD orientation map of transverse ground specimen of UNS S30403**

## A.3 Residual stress

In addition to EBSD characterization, it is important to be aware that different surface finishes can produce different residual stress values (Figure A.3). Thus, there is value in measuring residual stress to understand the different respond in environmentally assisted cracking tests or stress relieving, if that is relevant.

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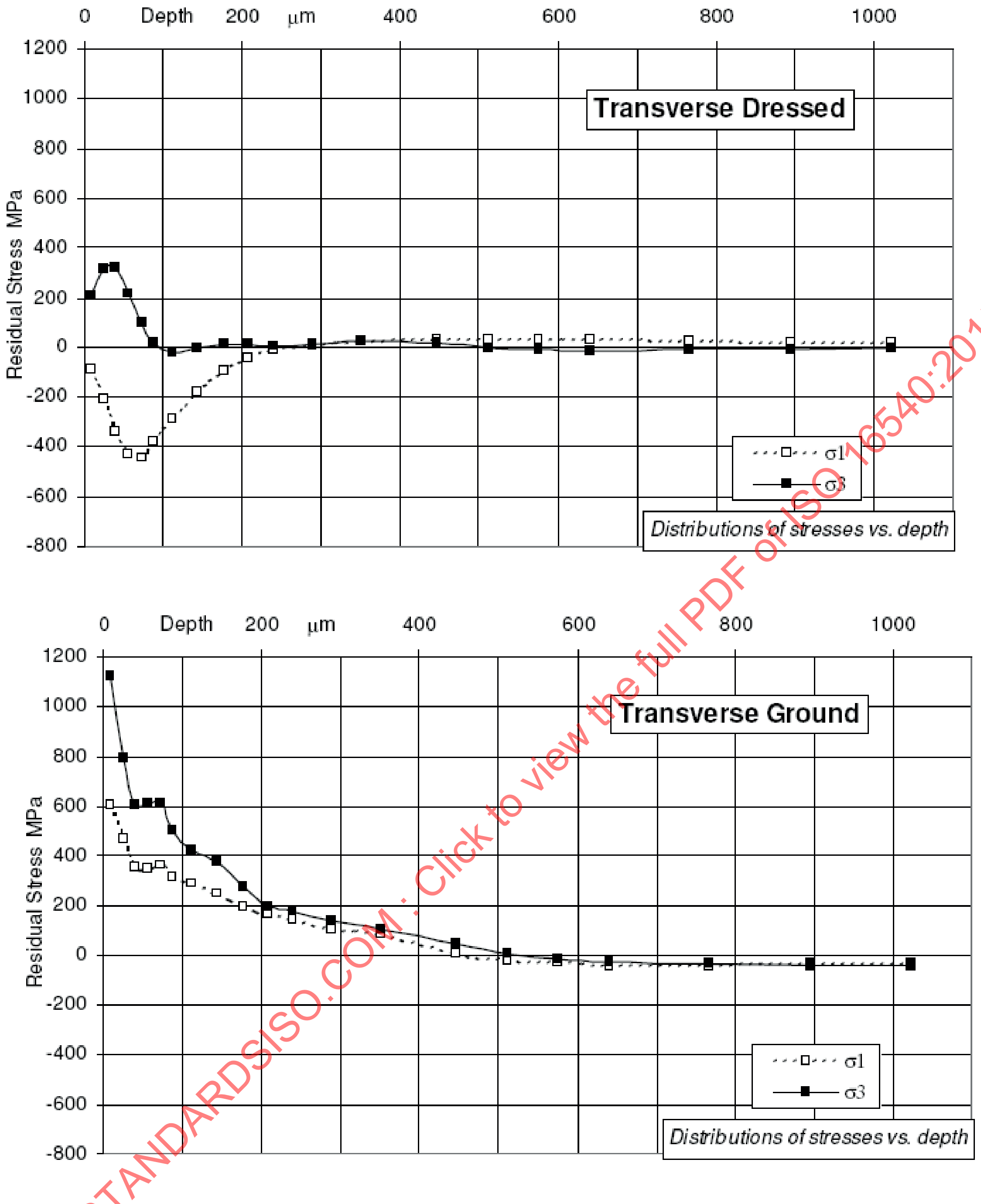


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**Figure A.3 — Examples of residual stress profiles for UNS S30403 with different surface finishes**

## A.4 Surface defects

In addition to near-surface deformation and residual stress, it is pertinent also to recognize that the machining/grinding process will generally introduce surface defects unless the surface is very finely ground or polished. An example is shown in Figure A.4. There is evidence for a greater propensity for corrosion damage to develop at such defects and it is important to be aware of their potential existence.


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