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Corrosion of metals and alloys — Stress corrosion testing —

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

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

Partie 9: Préparation et utilisation des éprouvettes préfissurées pour essais sous charge croissante ou sous déplacement croissant

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 7539-9 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

ISO 7539 consists of the following parts, under the general title *Corrosion of metals and alloys — Stress corrosion testing*:

— *Part 1: General guidance on testing procedures*

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

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

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

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

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

— *Part 7: 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*

Corrosion of metals and alloys — Stress corrosion testing —

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

1 Scope

1.1 This part of ISO 7539 covers procedures for designing, preparing and using pre-cracked specimens for investigating the susceptibility of metal to stress corrosion cracking by means of tests conducted under rising load or rising displacement. Tests conducted under constant load or constant displacement are dealt with in ISO 7539-6.

The term “metal” as used in this part of ISO 7539 includes alloys.

1.2 Because of the need to confine plasticity to the crack tip, pre-cracked specimens are not suitable for the evaluation of thin products such as sheet or wire and are generally used for thicker products including plate, bar and forgings. They can also be used for parts joined by welding.

1.3 Pre-cracked specimens may be stressed quantitatively with equipment for application of a monotonically increasing load or displacement at the loading points.

1.4 A particular advantage of pre-cracked specimens is that they allow data to be acquired from which critical defect sizes, above which stress corrosion cracking may occur, can be estimated for components of known geometry subjected to known stresses. They also enable rates of stress corrosion crack propagation to be determined.

1.5 A principal advantage of the test is that it takes into account the potential impact of dynamic straining on the threshold for stress corrosion cracking.

1.6 At sufficiently low loading rates, the K_{ISCC} determined by this method can be less than or equal to that obtained by constant load or displacement methods and can be determined more rapidly.

2 Normative references

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

ISO 7539-1:1987, *Corrosion of metals and alloys — Stress corrosion testing — Part 1: General guidance on testing procedures*

ISO 7539-6:—[1)], *Corrosion of metals and alloys — Stress corrosion testing —Part 6: Preparation and use of pre-cracked specimens for tests under constant load or constant displacement*

1) To be published. (Revision of ISO 7539-6:1989)

ISO 7539-7:—[2], *Corrosion of metals and alloys — Stress corrosion testing — Part 7: Slow strain rate testing*

ISO 11782-2:1998, *Corrosion of metals and alloys — Corrosion fatigue testing — Part 2: Crack propagation testing using precracked specimens*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 7539-6 as well as the following apply.

3.1
rate of change of crack opening displacement at loading plane
$\dot{V}_{LL}$
deflection at the loading point access measured over a fixed period

3.2
stress intensity factor at crack initiation
$K_{I\text{-init}}$
stress intensity applied at the commencement of measurable crack growth

3.3
range of stress intensity factor
ΔK_f**, in fatigue**
algebraic difference between the maximum and minimum stress intensity factors in a cycle

3.4
displacement rate
dq/dt
rate of increase of the deflection either measured at the loading point axis or away from the loading line

4 Principle

4.1 The use of pre-cracked specimens acknowledges the difficulty of ensuring that crack-like defects, introduced during either manufacture or subsequent service, are totally absent from structures. Furthermore, the presence of such defects can cause a susceptibility to stress corrosion cracking, which in some materials (e.g. titanium) may not be evident from tests on smooth specimens under constant load. The principles of linear elastic fracture mechanics can be used to quantify the stress situation existing at the crack tip in a pre-cracked specimen or structure in terms of the plane strain-stress intensity.

4.2 The test involves subjecting a specimen, in which a crack has been developed from a machined notch by fatigue, to an increasing load or displacement during exposure to a chemically aggressive environment. The objective is to quantify the conditions under which environmentally-assisted crack extension can occur in terms of the threshold stress intensity for stress corrosion cracking, K_{ISCC}, and the kinetics of crack propagation.

4.3 Tests may be conducted in tension or in bending. The most important characteristic of the test is the low loading/displacement rate that is applied.

4.4 Because of the dynamic straining which is associated with this method, the data obtained may differ from those obtained for pre-cracked specimens with the same combination of environment and material when the specimens are subjected to static loading only.

2) To be published. (Revision of ISO 7539-7:1989)

4.5 The empirical data can be used for design or life prediction purposes in order to ensure either that the stresses within large structures are insufficient to promote the initiation of environmentally-assisted cracking at whatever pre-existing defects may be present or that the amount of crack growth which would occur within the design life or inspection periods can be tolerated without the risk of unstable failure.

4.6 Stress corrosion cracking is influenced by both mechanical and electrochemical driving forces. The latter can vary with crack depth, opening or shape because of variations in crack-tip chemistry and electrode potential and may not be uniquely described by the fracture mechanics stress intensity factor.

4.7 The mechanical driving force includes both applied and residual stresses. The possible influence of the latter should be considered in both laboratory testing and application to more complex geometries. Gradients in residual stress in a specimen may result in non-uniform crack growth along the crack front.

4.8 K_{ISCC} is a function of the environment, which should simulate that in service, and of the conditions of loading.

5 Specimens

5.1 General

5.1.1 A wide range of standard specimen geometries of the type used in fracture toughness tests may be used. Those most commonly used are described in ISO 7539-6. The particular type of specimen used will be dependent upon the form, the strength and the susceptibility to stress corrosion cracking of the material to be tested and also on the objective of the test.

5.1.2 A basic requirement is that the dimensions be sufficient to maintain predominantly triaxial (plane strain) conditions in which plastic deformation is limited in the vicinity of the crack tip. Experience with fracture toughness testing has shown that for a valid K_{Ic} measurement, both the crack length, a, and the thickness, B, shall be not less than

$$2{,}5\left(\frac{K_{Ic}}{R_{p0,2}}\right)^2$$

and that, where possible, larger specimens where both a and B are at least

$$4\left(\frac{K_{Ic}}{R_{p0,2}}\right)^2$$

shall be used to ensure adequate constraint.

From the view of fracture mechanics, a minimum thickness from which an invariant value of K_{ISCC} is obtained cannot currently be specified. The presence of an aggressive environment during stress corrosion may reduce the extent of plasticity associated with fracture and hence the specimen dimensions needed to limit plastic deformation. However, in order to minimize the risk of inadequate constraint, it is recommended that similar criteria to those employed during fracture toughness testing used regarding specimen dimensions, i.e. both a and B shall be not less than

$$2{,}5\left(\frac{K_{I}}{R_{p0,2}}\right)^2$$

and preferably shall be not less than

$$4\left(\frac{K_\mathrm{I}}{R_{\mathrm{p0,2}}}\right)^2$$

where K_I is the stress intensity to be applied during testing, in MPa/m.

As a test for its validity, the threshold stress intensity value eventually determined shall be substituted for K_I in the first of these expressions.

5.1.3 If the specimens are to be used for the determination of K_{ISCC}, the initial specimen size shall be based on an estimate of the K_{ISCC} of the material (in the first instance, it being better to over-estimate the K_{ISCC} value and therefore use a larger specimen than may eventually be found necessary). Where the service application involves the use of material of insufficient thickness to satisfy the conditions for validity, it is permissible to test specimens of similar thickness, provided that it is clearly stated that the threshold intensity value obtained, K_{QSCC}, is of relevance only to that specific application. Where it is required to determine stress corrosion crack growth behaviour as a function of stress intensity, the specimen size should be based on an estimate of the highest stress intensity at which crack growth rates are to be measured.

5.1.4 A wide choice of specimen geometries is available to suit the form of the test material, the experimental facilities available and the objectives of the test. Two basic types of specimen can be used

a) those intended for being loaded by means of a tensile force;

b) those intended for being loaded by means of a bending force.

This means that crack growth can be studied under either bend or tension loading conditions. The specimens can be used for either the determination of K_{ISCC} by the initiation of a stress corrosion crack from a pre-existing fatigue crack using a series of specimens and for measurements of crack growth rates. Since the specimens are loaded during exposure to the test environment, the risk of unnecessary incubation periods is avoided.

5.1.5 Crack length measurements can be readily made with a number of continuous monitoring methods such as the electrical resistance technique.

5.1.6 Bend specimens can in principle be tested in relatively simple cantilever beam equipment but specimens subjected to tension loading require a tensile test machine.

5.2 Specimen design

5.2.1 The specimens can be subjected to either tension or bend loading. Depending on the design, tension loaded specimens can experience stresses at the crack tip which are predominantly tensile (as in remote tension types such as the centre-cracked plate) or contain a significant bend component (as in crackline loaded types such as compact tension specimens). The presence of significant bending stress at the crack tip can adversely affect the crack path stability during stress corrosion testing and can facilitate crack branching in certain materials. Bend specimens can be loaded in 3-point, 4-point or cantilever bend fixtures.

5.2.2 The occurrence of crackline bending with an associated tendency for crack growth out of plane can be curbed by the use of side grooves.

5.2.3 A number of specimen geometries have specific advantages, which have caused them to be frequently used for rising load/displacement stress corrosion testing. These include:

a) compact tension (CTS) specimens, which minimize the material requirement;

b) cantilever bend specimens, which are easy to machine and inexpensive to test;

c) C-shaped specimens, which can be machined from thick walled cylinders in order to study the radial propagation of longitudinally oriented cracks.

Details of standard specimen designs for each of these types of specimen are given in Figures 1 to 3.

5.2.4 If required, e.g. if fatigue crack initiation and/or propagation is difficult to control satisfactorily, a chevron notch configuration as shown in Figure 4 may be used. If required, its included angle may be increased from 90° to 120°.

5.2.5 Where it is necessary to measure crack opening displacements, knife edges for the location of displacement gauges can be machined into the mouth of the notch, as shown in Figure 5a). Alternatively, separate knife edges can either be screwed or glued on to the specimen at opposite sides of the notch, as shown in Figure 5b). Details of a suitable tapered beam displacement gauge are given in Figure 6.

5.3 Stress intensity factor considerations

5.3.1 It can be shown, using elastic theory, that the stress intensity, K_I, acting at the tip of a crack in specimens or structures of various geometries can be expressed by relationships of the form

$$K_I = Q \times \sigma \times \sqrt{a}$$

where

Q is a dimensionless geometrical constant;

σ is the applied stress in MPa;

a is the crack length in metres.

5.3.2 The solutions for K_I for specimens of particular geometry and loading method can be established by means of finite element stress analysis, or by either experimental or theoretical determinations of specimen compliance.

5.3.3 K_I values can be calculated by means of a dimensionless stress intensity coefficient, Y, related to crack length expressed in terms of a/W through relationship of the form

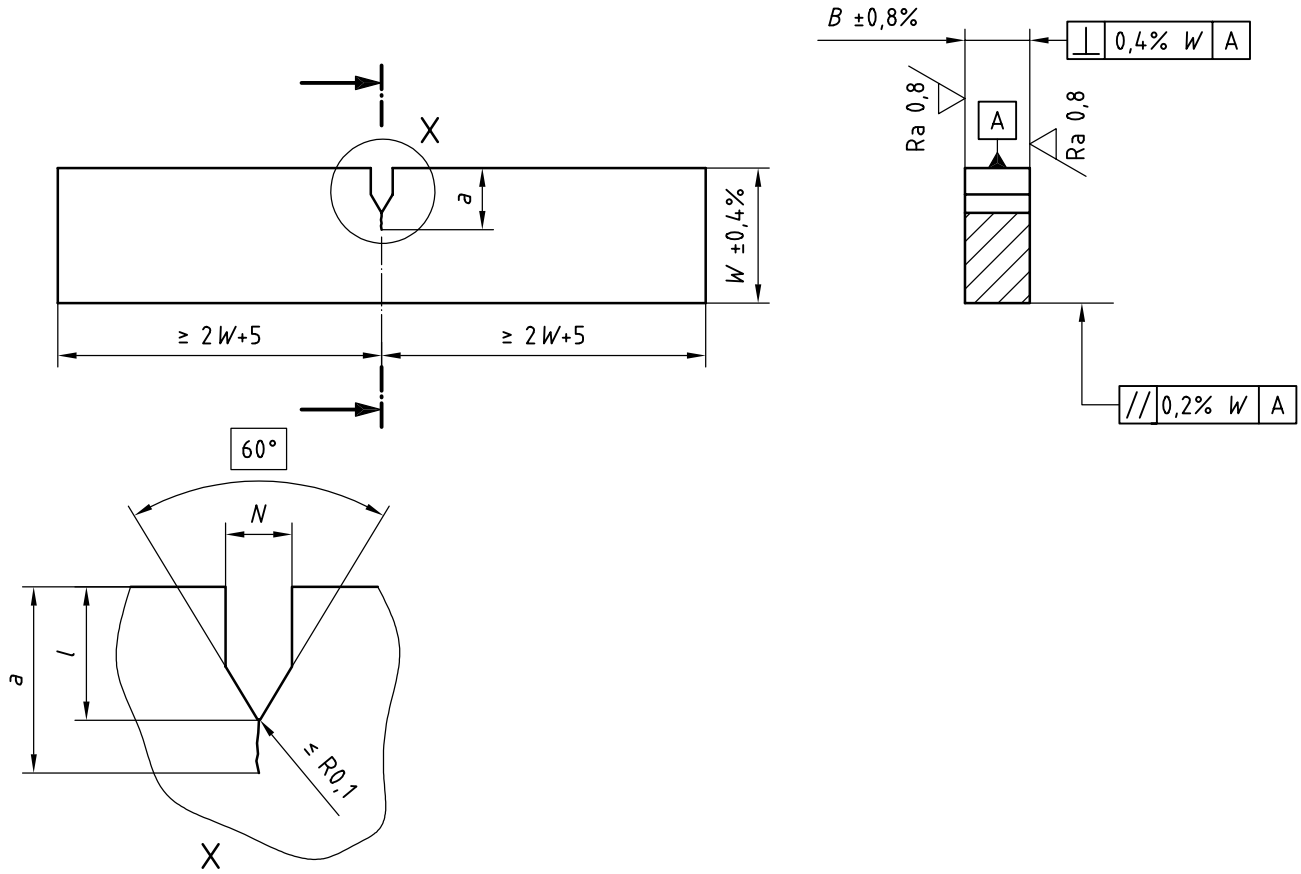
$$K_I = \frac{YP}{B\sqrt{W}}$$

for compact tension and C-shaped specimens, where W is the width of the specimen in metres and P the applied load.

5.3.4 Where it is necessary to use side-grooved specimens in order to curb crack branching tendencies, etc., shallow side grooves (usually 5 % of the specimen thickness on both sides) can be used. Either semi-circular or 60° V-grooves can be used, but it should be noted that even with semi-circular side grooves of up to 50 % of the specimen thickness, it is not always possible to maintain the crack in the desired plane of extension. Where side grooves are used, the effect of the reduced thickness, B_n, due to the grooves on the stress intensity can be taken into account by replacing B with $\sqrt{BB_n}$ in the above expression. However, the influence of side grooving on the stress intensity factor is far from established and correction factors should be treated with caution, particularly if deep side grooves are used.

5.3.5 Solutions for Y for specimens with geometries which are often used for stress corrosion testing are given in Figures 7 to 9.

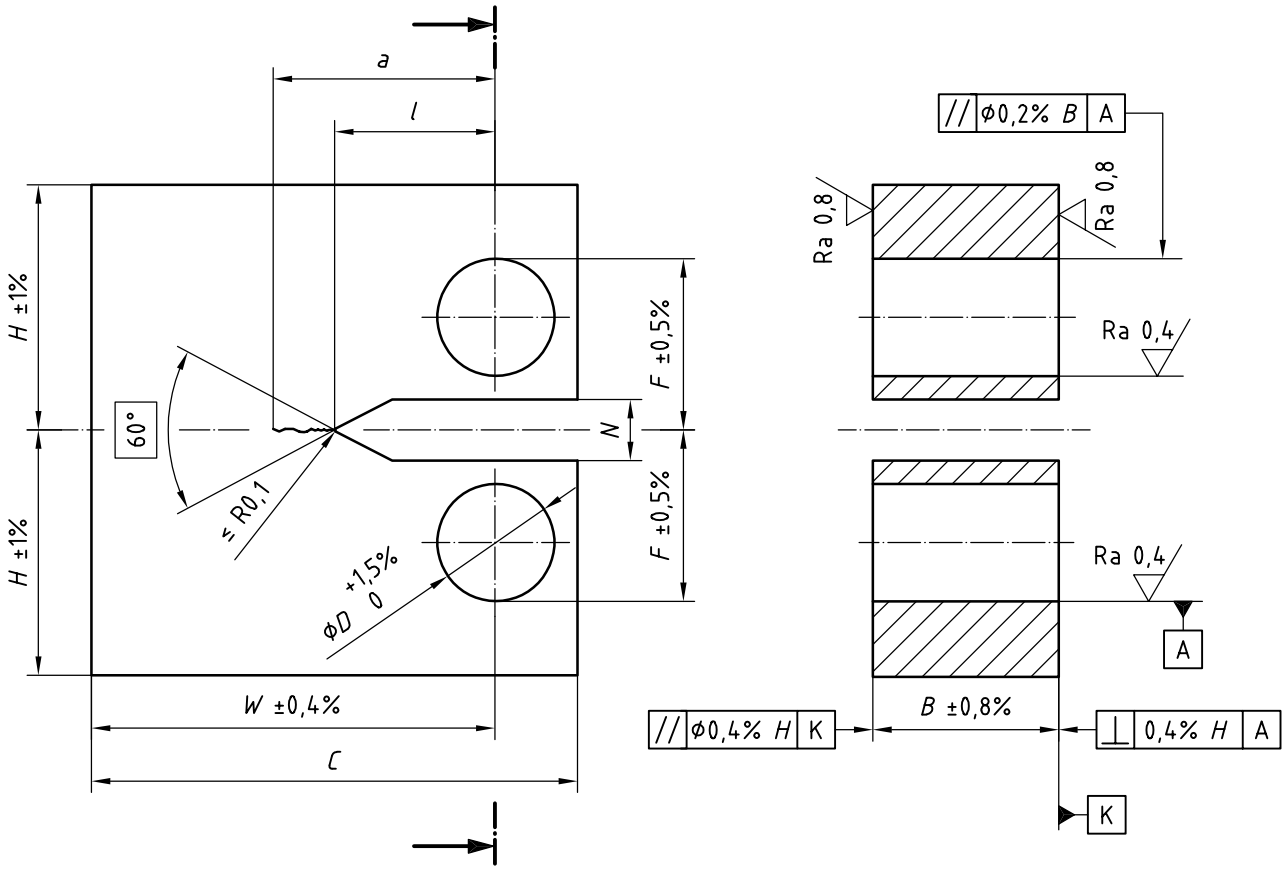
Dimensions in millimetres



Width	= W
Thickness, B	= 0,5 W
Notch width, N	= 0,065 W maximum (if $W > 25$ mm) or 1,5 mm maximum (if $W \leqslant 25$ mm)
Effective notch length, l	= 0,25 W to 0,45 W
Effective crack length, a	= 0,45 W to 0,55 W

Figure 1 — Proportional dimensions and tolerances for cantilever bend test pieces

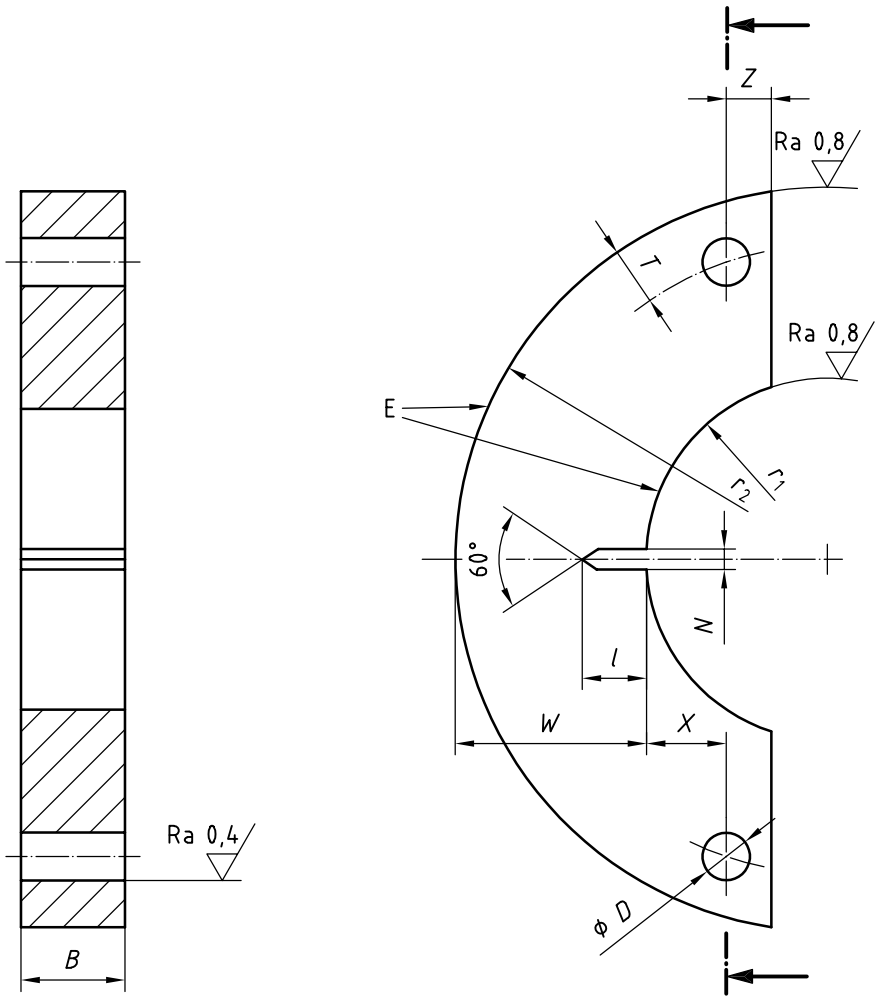
Dimensions in millimetres



Net width	= W
Total width, C	= 1,25 W minimum
Thickness, B	= 0,5 W
Half height, H	= 0,6 W
Hole diameter, D	= 0,25 W
Half distance between hole outer edges, F	= 1,6 D
Notch width, N	= 0,065 W maximum
Effective notch length, l	= 0,25 W to 0,40 W
Effective crack length, a	= 0,45 W to 0,55 W

Figure 2 — Proportional dimensions and tolerances for compact tension test pieces

Dimensions in millimetres

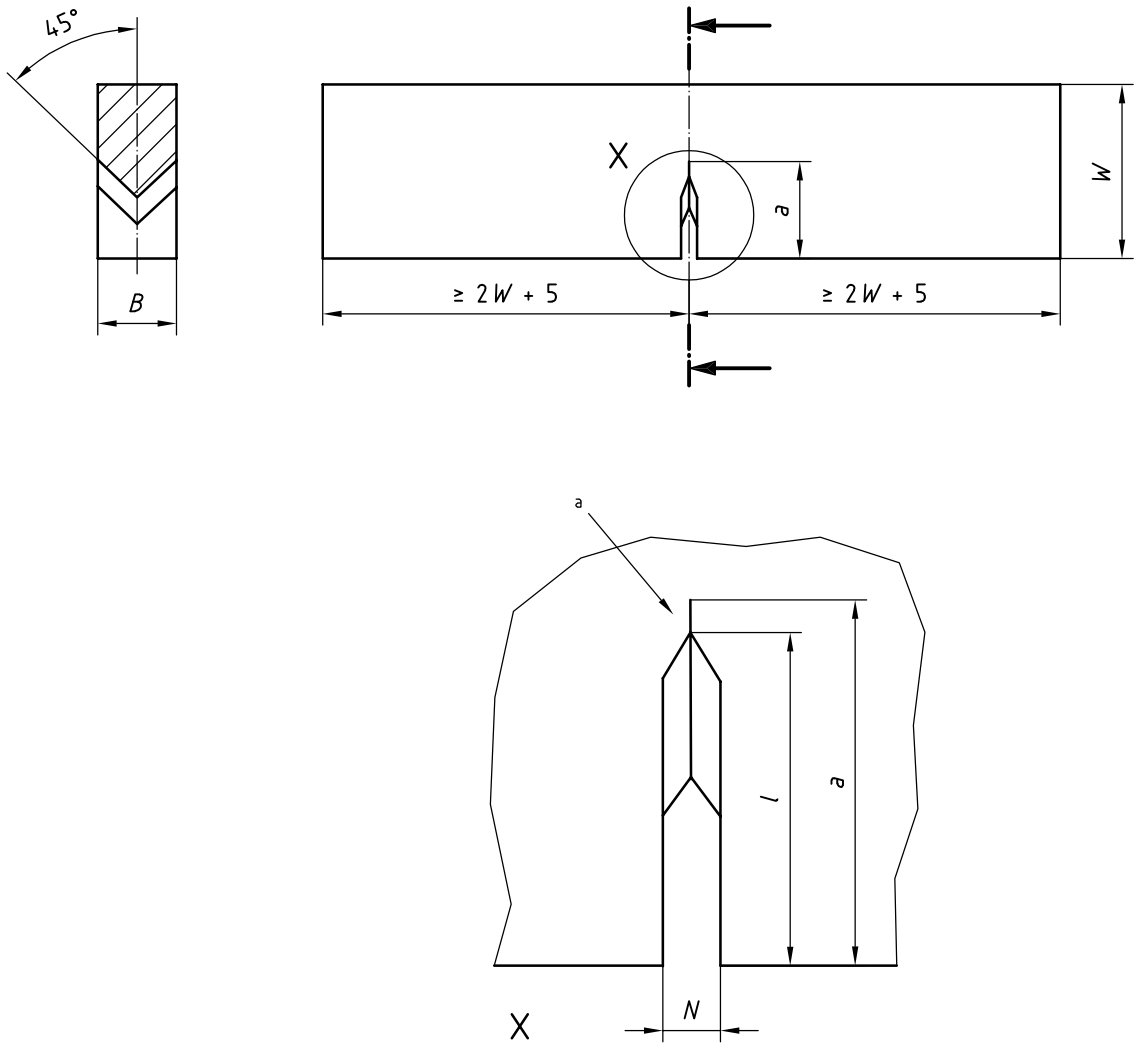


Net width	= W
Thickness B	= 0,50 W ± 0,01 W
Axis of holes to tangent to inner radius, X	= 0,50 W ± 0,005 W
Notch width, N	= 1,5 mm minimum (0,1 W maximum)
Notch width, I	= 0,3 W
Axis of holes to face of specimen, Z	= 0,25 W ± 0,01 W
Axis of holes toouter surface, T	= 0,25 W ± 0,01 W
Diameter of holes, D	= 0,25 W ± 0,005 W

NOTE All surfaces should be perpendicular and parallel, as applicable, to within 0,002 W TIR and “E” surfaces perpendicular to “Y” surfaces to within 0,02 W TIR:

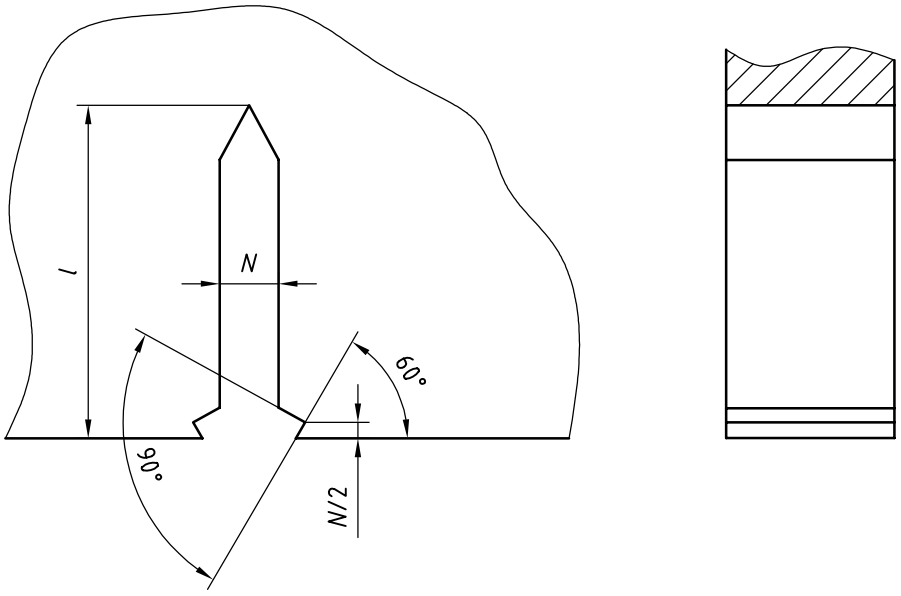
Figure 3 — Proportional dimensions and tolerances for C-shaped test pieces

Dimensions in millimetres

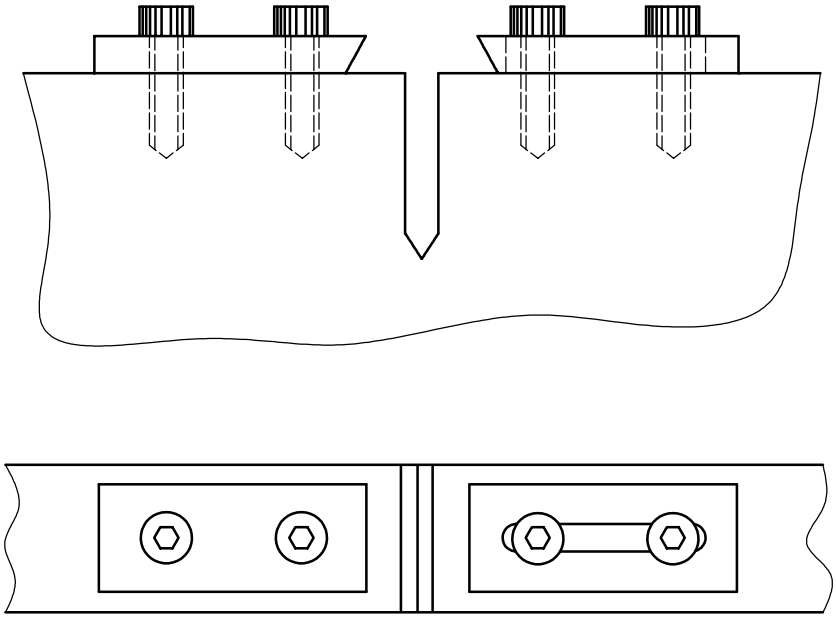


a Mill with 60° cutter, notch root radius 0,3 maximum for all test piece sizes.

Figure 4 — Chevron notch



a) Integral type

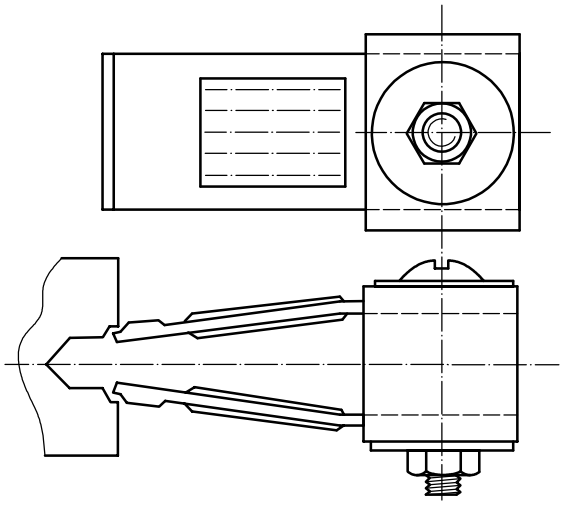


b) Screw-on type

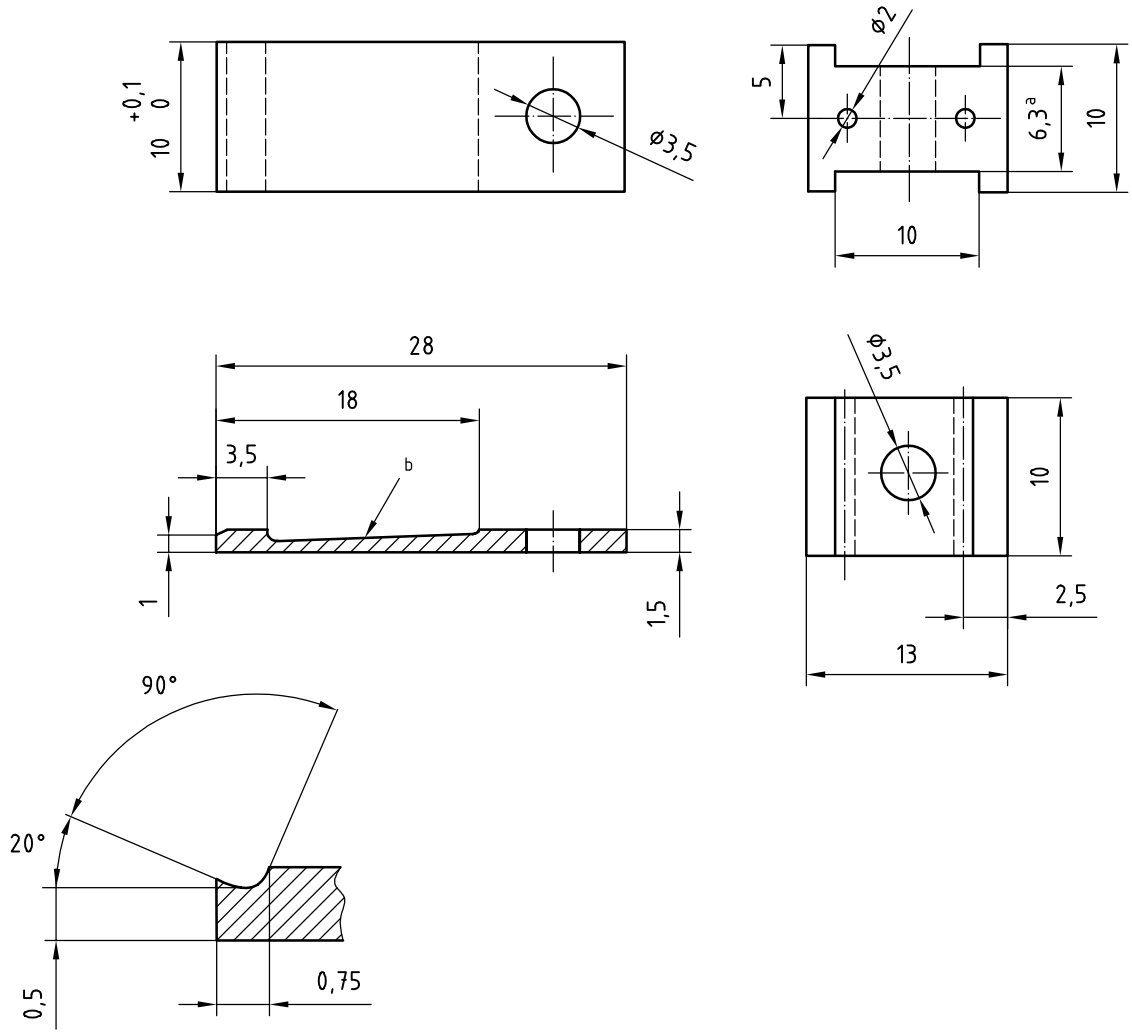
NOTE Provided adequate strength can be assured, the above knife edges may be fixed using adhesive.

Figure 5 — Knife edges for location of displacement gauges

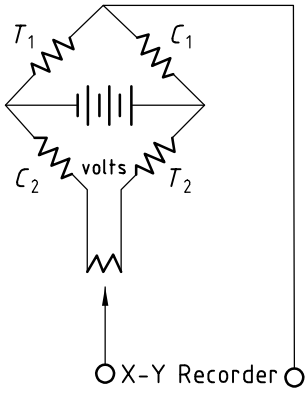
Dimensions in millimetres



a) Displacement gauge mounted on a test piece



b) Dimensions of beams



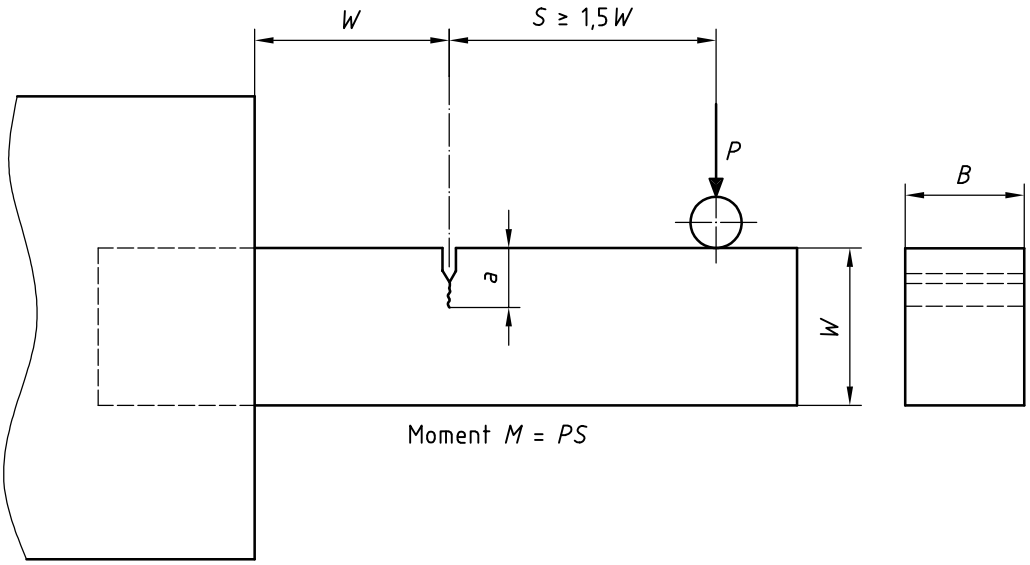
c) Bridge measurement circuit

[a] This dimension should be 3,8 × the minimum initial gauge length

[b] Beam thickness taper 0,5 to 0,8

NOTE Strain gauges and materials should be selected to suit the test environment.

Figure 6 — Details of tapered beam displacement gauge

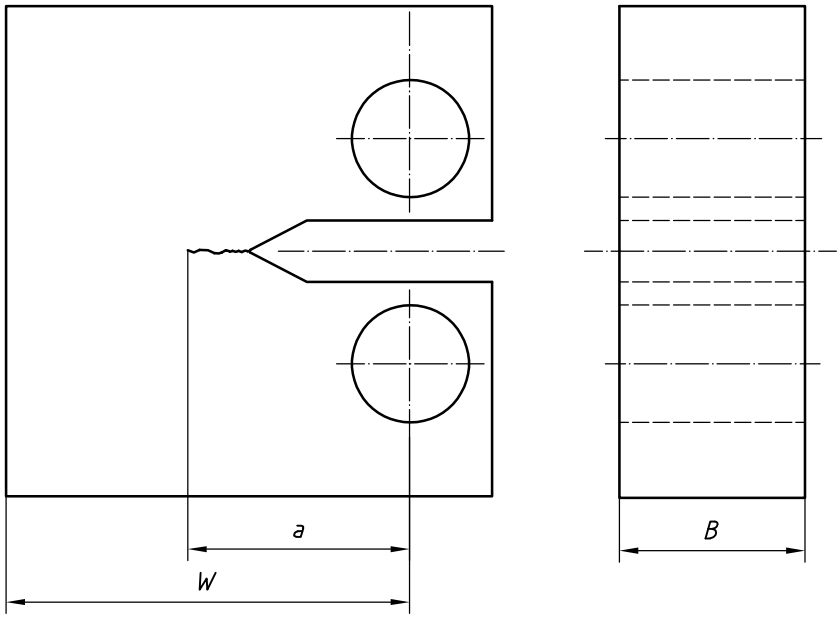


$$K_I = \frac{YP}{B\sqrt{W}}$$

where $Y = 6,21\sqrt{\frac{1}{\sqrt{\left(1-\frac{a}{W}\right)^3}} - \left(1+\frac{a}{W}\right)^3}$ in the case where S = 1,5 W

NOTE This expression was originally derived from the combined techniques of stress analysis and compliance and although its inaccuracy and validity limits are not well-defined, it has been used over the range $0,2 \leqslant \frac{a}{W} \leqslant 0,6$. For greatest confidence, it is recommended that an emprical compliance be used.

Figure 7 — Stress intensity solution for cantilever bend specimen

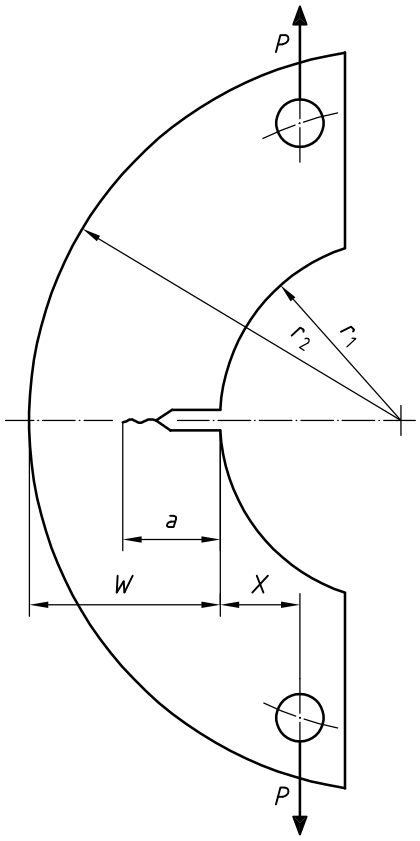


$$K_I = \frac{YP}{B\sqrt{W}}$$

where $$Y = \frac{2+\frac{a}{W}}{\sqrt{\left(1-\frac{a}{W}\right)^3}}\left[0{,}886 + 4{,}64\left(\frac{a}{W}\right) - 13{,}32\left(\frac{a}{W}\right)^2 + 14{,}72\left(\frac{a}{W}\right)^3 - 5{,}6\left(\frac{a}{W}\right)^4\right]$$

NOTE The inaccuracy of this expression is considered to be no greater than ± 0,5 % over the range $0{,}2 \leqslant \frac{a}{W} \leqslant 1{,}0$.

Figure 8 — Stress intensity solution for compact tension specimen



$$K_{\mathrm{I}} = \frac{YP}{B\sqrt{W}}$$

where $Y = \left(18{,}23\sqrt{\frac{a}{W}} - 106{,}2\sqrt{\frac{a^3}{W}} + 397{,}7\sqrt{\frac{a^5}{W}} - 582{,}0\sqrt{\frac{a^7}{W}}\ 369{,}1\sqrt{\frac{a^9}{W}}\right) \times \left(1 + 1{,}54\,\frac{X}{W} + 0{,}5\frac{a}{W}\right) \times \left[1 + 0{,}22\left(1 - \sqrt{\frac{a}{W}}\right)\left(1 - \frac{r_1}{r_2}\right)\right]$

NOTE The inaccuracy of this expression is considered to be no greater than 1 % over the range $0{,}45 \leqslant \frac{a}{W} \leqslant 0{,}55$. However, it can be used over the wider range $0{,}3 \leqslant \frac{a}{W} \leqslant 0{,}7$ when $0 \leqslant \frac{X}{W} \leqslant 0{,}7$ and $0 \leqslant \frac{r_1}{r_2} \leqslant 1$ in which case the accuracy is believed to be no greater than 2 %.

Figure 9 — Stress intensity solution for C-shaped specimen

5.4 Specimen preparation

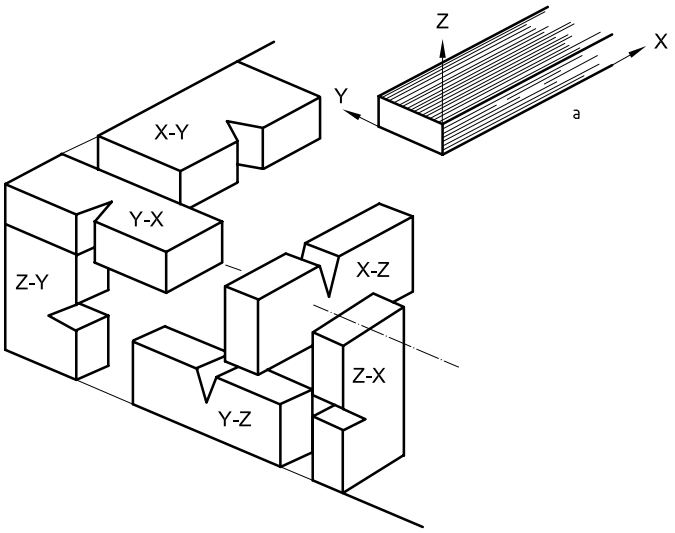
5.4.1 Residual stresses can have an influence on stress corrosion cracking. The effect can be significant when test specimens are removed from material in which complete stress relief is impractical, such as weldments, as-quenched materials and complex forged or extruded shapes. Residual stresses superimposed on the applied stress can cause the localized crack-tip stress intensity factor to be different from that computed solely from externally applied loads. The presence of significant residual stress, often in the form of irregular crack growth, namely excessive crack front curvature or out-of-plane crack growth, generally indicates that residual stresses are affecting behaviour. Measurement of residual stress is desirable.

5.4.2 Specimens of the required orientation (see Figure 10) shall, where possible, be machined in the fully heat-treated condition. For specimens in material that cannot easily be completely machined in the fully heat-treated condition, the final heat treatment may be given prior to the notching and finishing operations provided that at least 0,5 mm per face is removed from the thickness at this finish machining stage. However, heat treatment may be carried out on fully machined specimens in cases in which heat treatment will not result in detrimental surface conditions, residual stress, quench cracking or distortion.

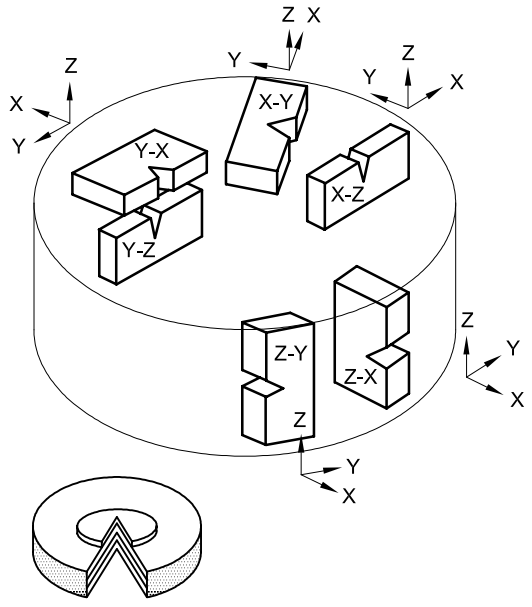
5.4.3 After machining, the specimens shall be fully degreased in order to ensure that no contamination of the crack tip occurs during subsequent fatigue pre-cracking or stress corrosion testing. In cases where it is necessary to attach electrodes to the specimen by soldering or brazing for crack monitoring by means of electrical resistance measurements, the specimens shall be fully degreased following this operation prior to pre-cracking in order to remove traces of remnant flux.

5.5 Specimen identification

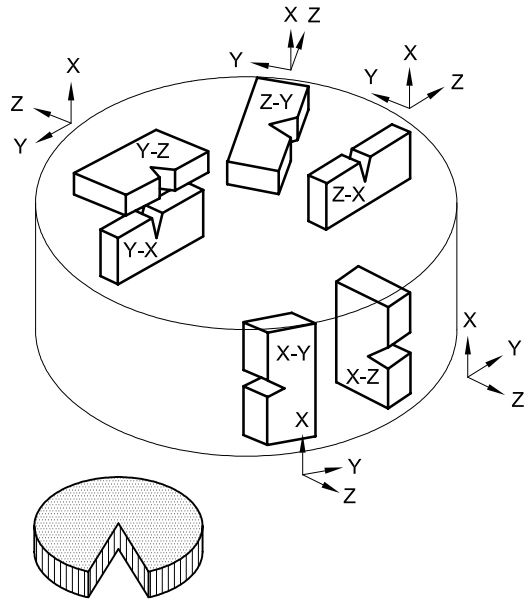
Specimen identification marks may be stamped or scribed on either the face of the specimen bearing the notch or on the end faces parallel to the notch.



a) Basic fracture plane identification: rectangular section

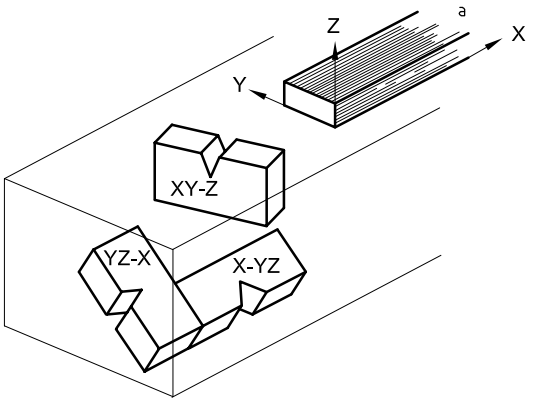


1) Radial grain flow — Axial working direction



2) Axial grain flow — Radial working direction

b) Basic fracture plane identification: cylindrical sections



c) Non-basic fracture plane identification

[a] Grain flow

Figure 10 — Fracture plane identification

6 Initiation and propagation of fatigue cracks

6.1 The machine used for fatigue cracking shall have a method of loading such that the stress distribution is symmetrical about the notch and the inaccuracy in measurement of applied load is no greater than $\pm$ 2,5 %.

6.2 The environmental conditions applied during fatigue pre-cracking, as well as the stressing conditions, can influence the subsequent behaviour of the specimen during stress corrosion testing. In some materials, the introduction of the stress corrosion test environment during the pre-cracking operation promotes a change from the normal ductile transgranular mode of fatigue cracking to one which more closely resembles stress corrosion cracking. This may facilitate the subsequent initiation of stress corrosion cracking and lead to the determination of conservative initiation values of K_{ISCC}. However, unless facilities are available to commence stress corrosion testing immediately following the pre-cracking operation, corrodant remaining at the crack tip may promote blunting due to corrosive attack. Furthermore, the repeatability of results may suffer when pre-cracking is conducted in the presence of an aggressive environment, because of the greater sensitivity of the corrosion fatigue fracture mode to the cyclic loading conditions. In addition, more elaborate facilities may be needed for environmental control purposes during pre-cracking. For these reasons, it is recommended that, unless agreed otherwise between the parties, fatigue pre-cracking be conducted in the normal laboratory air environment.

6.3 The specimens shall be pre-cracked by fatigue loading with an R value in the range 0 to 0,1 until the crack extends at least 2,5 % W or 1,25 mm beyond the notch at the side surfaces, whichever is greater. The crack may be started at higher K_{I} values but, during the final 0,5 mm of crack extension, the fatigue pre-cracking shall be completed at as low a maximum stress intensity as possible (below the expected K_{ISCC}).

NOTE Load shedding procedures as described in ISO 11782-2 may be helpful when the K_{ISCC} values are expected to be low.

6.4 The final length of the fatigue crack shall be such that the requirement for plane strain predominance is satisfied, i.e.

$$a \geqslant 2{,}5\left(\frac{K_{\mathrm{I}}}{R_{\mathrm{p0,2}}}\right)^2$$

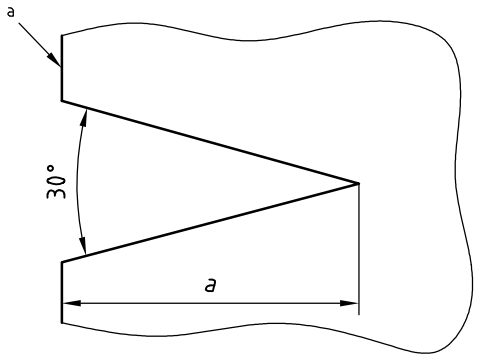
This condition is optimized when the final a/W ratio is in the range 0,45 to 0,55.

NOTE Crack size may be important in relation to SCC.

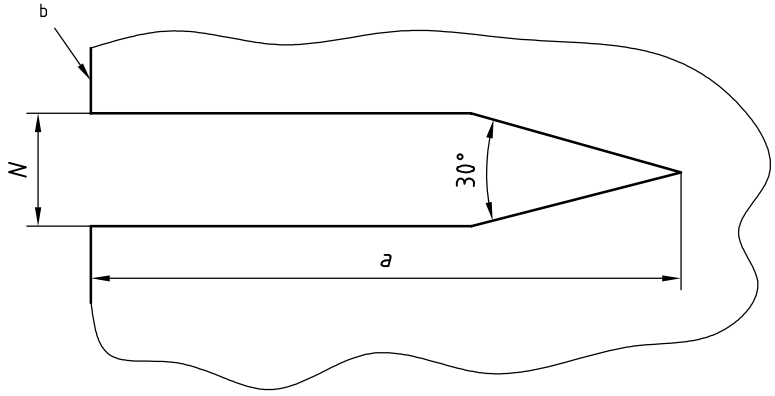
6.5 In order to avoid interaction of the stress field associated with the crack with that due to the notch, the crack shall lie within the limiting envelope as shown in Figure 11, in which examples for bend and tensile pieces are shown. For the example valid for bend or tensile test pieces, if the apex of the envelope is located at the tip of the fatigue crack, the whole of the machined notch shall lie within the envelope as is shown in Figure 11 c).

6.6 In order to ensure the validity of the stress intensity analysis, the fatigue crack shall be inspected on each side of the specimen to ensure that no part of it lies in a plane the slope of which exceeds an angle of 10° from the plane of the notch and that the difference in lengths does not exceed 5 % W.

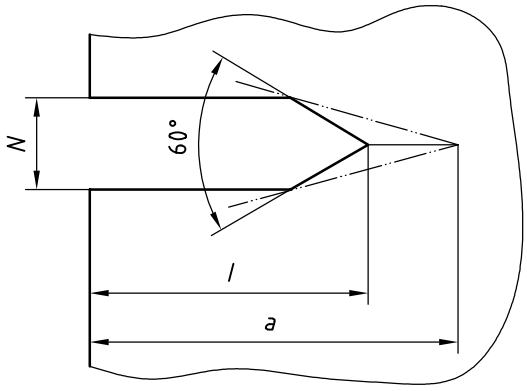
6.7 Additional guidance on fatigue pre-cracking procedures is available in ISO 11782-2.



a) Bend test piece



b) Tensile test piece



c) Bend or tensile test piece

a Edge of test piece

b Loading line of test piece

Figure 11 — Envelope limiting size and form of notch and fatigue crack

7 Procedure

7.1 General

Before testing, the thickness B and width W shall be measured to within 0,1 % W on a line no further than 10 % W from the crack plane. The average length of the fatigue pre-crack on both sides of the specimen shall also be determined and this value used in assessing the pre-load required to produce the desired initial stress intensity, K_I (see 7.6).

7.2 Environmental considerations

7.2.1 Because of the specificity of metal-environment interactions, it is essential that stress corrosion crack propagation tests be conducted under environmental conditions that are closely controlled (see 7.2.3 and 7.2.4).

7.2.2 The environmental testing conditions depend upon the intent of the test but, ideally, should be the same as those prevailing for the intended use of the alloy or comparable to the anticipated service condition.

7.2.3 Environmental factors of importance are electrode potential, temperature, solution composition, pH, concentration of dissolved gases, flowrate and pressure. ISO 7539-1 provides useful background information. In relation to gaseous environments a critical factor is purity of the gas.

7.2.4 Tests may be conducted under open circuit conditions in which the electrode potential of the metal is dependent on the specific environmental conditions of the test, of which the degree of aeration is an important factor. Alternatively, the electrode potential may be displaced from the open circuit value by potentiostatic or galvanostatic methods.

7.2.5 Auxiliary electrodes to apply external current shall be designed to produce uniform current distribution on the specimen, i.e. the electrode potential shall be constant.

7.2.6 When practical, it is recommended that the specimens be stressed after being brought into contact with the test environment. Otherwise, the stressed specimens shall be exposed to the test environment as soon as possible after stressing.

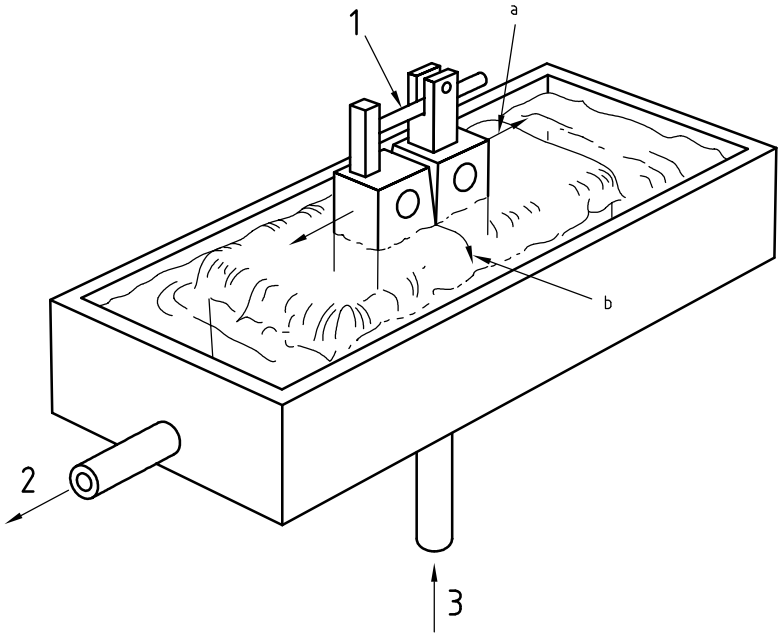
7.3 Environmental chamber

7.3.1 The environmental chamber shall completely enclose the test section of the specimen. Wherever possible, the gripped portions shall be excluded from contact with the solution environment in order to prevent galvanic effects and crevice corrosion. These problems can be overcome by the use of a local environmental cell of the type shown in Figure 12 in which the environment is circulated around the vicinity of the notch, pre-crack and anticipated crack growth region of the specimen. Crevice problems may also arise where the specimen emerges from the test cell and these can be avoided by appropriate design of the cell or by the use of protective coatings at such locations. If total immersion in the corrodent is contemplated, the loading points shall be protected against corrosion. If this is not possible, appropriate measures shall be taken through, e.g., the use of similar metals, electrical insulation or coatings.

7.3.2 An adequate volume-of-solution:metal-area ratio is required (dependent on reaction rates and exposure time) and a circulation system is usually necessary. For conditions of applied potential or applied current, a separate compartment for the counter electrode may be necessary in order to limit any effects caused by reaction products from this electrode. It should be noted that potentiostatic control at the tip of a stress corrosion crack may be subject to large variations as the crack length increases, which must be taken into account when considering mechanisms of stress corrosion cracking.

7.3.3 Non-metallic materials are recommended for the environmental chamber and circulation system where this is practicable. These materials shall be inert. Note that glass and certain plastics are not inert at elevated temperatures. Where metallic chambers are necessary, these shall be electrically insulated from the specimen in order to prevent galvanic interaction.

7.3.4 For tests in a gaseous environment, an all-metal-chamber is preferred.



Key

1 displacement transducer
2 solution outlet
3 solution inlet

[a] Load
[b] Solution flow

Figure 12 — Position of environmental cell on a fracture mechanics specimen

7.4 Environmental control and monitoring

7.4.1 The environment shall be monitored and controlled during the test as required. In unbuffered systems, the pH can be maintained constant using an automatic pH control system; otherwise the effect of any variations in pH on crack growth shall be assessed.

7.4.2 In systems open to the atmosphere, aeration can be maintained by bubbling air through the solution. In closed systems, monitoring is required. The flowrates used in testing shall simulate the range of conditions in service because flow can affect the electrode potential, e.g. by influencing the flux of oxygen and mass transfer between the crack enclave and the bulk solution. The orientation of flow with respect to the crack can be important in the latter case. Sealing of the crack sides to limit artificial through-thickness transport should be considered but may introduce local crevice problems.

7.4.3 It is strongly recommended that the electrode potential be measured with a reference electrode appropriate for the application. Care shall be taken to limit IR drop in the measurement of potential. The temperature of the solution shall be controlled to within $\pm$ 2 °C of the reported value.

7.5 Selection of initial K value prior to dynamic loading

7.5.1 In cases where data from fracture toughness tests in air for the material under investigation are not available, a preliminary test shall be performed in laboratory air. This requires that an initial specimen be used to determine the fracture toughness of the material, K_{Ic} (or K_Q if invalid), using recommended procedures. This value establishes the upper limit of K_{ISCC}.

7.5.2 The establishment of cracking conditions in a given metal/environment combination may be time-dependent if they do not exist at the outset of the test. In such circumstances, stress corrosion cracking may only be observed if the displacement rate is sufficiently low to ensure that failure due to pure mechanical rupture does not occur before the necessary time has elapsed, whereby the necessary environmental conditions for cracking have been established. These difficulties can sometimes be minimized by exposure of the specimens to the test environment for a period prior to the initiation of dynamic strain. It is recommended to keep the specimens under pre-load for a period of at least 24 h in the test environment before starting the test. A typical pre-load for the first test in environment is a value which corresponds to an initial K value of 5 % of K_{Ic} (or K_Q).

7.5.3 Selection of initial K value is important because it determines the length of the test.

7.5.4 The initial value of K may correspond to the final K_{max} following fatigue pre-cracking. Where K_{ISCC} is considered likely to be high, the load may be stepped up to an arbitrary higher value prior to dynamic loading. If cracking subsequently ensues without a subsequent increase in K value, a lower initial K shall be chosen. Since dynamic loading usually represents an accelerated test procedure compared to static loading, a low initial K can be adopted. However, the choice of initial K value may be refined based on the first estimate of $K_{I\text{-}init}$.

7.6 Determination of K_{ISCC}

7.6.1 General

The tests are performed at displacement rates which are selected prior to each test and which shall be kept constant throughout this test.

7.6.2 Determination schedule

7.6.2.1 In most systems, the stress intensity factor at crack initiation, $K_{I\text{-}init}$, is likely to be a function of the applied displacement rate. Therefore, tests shall be conducted over an appropriate range of displacement rates for the system under consideration in order to ensure that a conservative value of K_{ISCC} is obtained. The procedure to be adopted involves subjecting a number of specimens to different displacement rates following the schedule outlined in 7.6.2.2 to 7.6.2.9.

7.6.2.2 An arbitrary but low displacement rate shall be chosen for the determination of a preliminary $K_{I\text{-}init}$ value above which stress corrosion cracking is likely to initiate. This rate depends upon the material and environment in question and shall be agreed between parties concerned although for preliminary testing, rates of 1×10^{-8} m/s (36 µm/h) for titanium alloys and 1×10^{-9} m/s (3,6 µm/h) for higher strength steels and aluminium alloys may be appropriate. Some recommendations for determining an appropriate initial displacement rate are given in Annex A.

The test is started from the pre-load chosen in section 7.5.2, without unloading the specimen.

7.6.2.3 During testing, crack length may be monitored continuously by means of electrical resistance, back face strain, or alternative techniques, depending on the experimental circumstances. These measurements shall enable the detection of crack initiation. They also enable crack growth rates to be determined as a function of stress intensity factors.

7.6.2.4 The load-displacement behaviour of the specimen and the time elapsed since the start of the test are measured and recorded. The test machine shall be stopped after either the indirect crack length measuring method or a drop in load record indicates that crack initiation and subsequent crack growth have occurred.

7.6.2.5 On completion of the test, the specimen is taken out of the test environment and the crack front marked by either heat tinting or fatigue cracking in air. The fatigue cracking shall be performed at an R ratio greater than 0,6 in order to avoid damage to the fracture surfaces from crack closure effects. The maximum fatigue load shall not exceed three quarters of the final load measured during the test. In certain environments, it may be apparent that the crack front at test termination is already sufficiently marked by visible remains of the environment so that no additional marking is required.

7.6.2.6 The specimen shall be broken and the length of the fatigue pre-crack measured at both edges and at the following three positions: 0,25 B; 0,50 B and 0,75 B.

The average of these five measurements shall be used as the effective initial crack length a_0 in the calculation of $K_{\text{I-init}}$.

The test is invalid if

a) the difference between any two of these last three measurements exceeds 2,5 % W;

b) the difference between the maximum and minimum crack lengths exceeds 5 % W;

c) any part of the fatigue crack surface lies in a plane the slope of which exceeds an angle of 10° from the plane of the notch:

d) the fatigue crack is not in one plane, i.e. effects of multi-nucleation are present;

e) the factor $2{,}5\left(\frac{K_{\text{I-init}}}{R_{\text{p0,2}}}\right)^2$ is greater than the thickness of the specimen and/or the crack length;

f) there is uncertainty over the fatigue crack length.

7.6.2.7 A new specimen is tested under identical environmental conditions at a lower displacement rate. The initial K value may be modified.

NOTE Guidance on selection of the new displacement rate can be made if the fracture surface is examined by means of microscopy, for evidence of stress corrosion crack extension in comparison with the fracture surface of a specimen that was tested in air: The percentage of environmental cracking on the fracture surface in the region of stable crack extension adjacent to the initial crack front can be used as an estimate of a suitable displacement rate for the subsequent test according to Table 1.

If the whole region of stable crack extension is covered by environmental cracking, then a further reduction of displacement rate may not be necessary, although advisable for confirmation. Otherwise, a displacement rate shall be selected which, by a factor of 10, is lower than the rate chosen for the preceding test.

Table 1 — Recommended factors by which the strain rate shall be reduced depending upon the proportion of stress corrosion cracking on the fracture surface

Proportion of SCC on fracture surface, %	Factor by which the displacement rate shall be reduced
< 10	50
10 to 30	20
30 to 50	10
50 to 80	5
> 80	2

7.6.2.8 If the values of $K_{\text{I-init}}$ determined from two subsequent tests do not differ by more than 5 % (or the desired accuracy of the K_{ISCC} value), then the lower of these two values can be considered as the preliminary value of K_{QSCC}.

7.6.2.9 If time permits, the reliability of the preliminary value of K_{QSCC} can be checked by a further stress corrosion test at a displacement rate which is by a factor of 5 to 10 lower than the one used in step 7.6.2.8. Further testing will only be necessary if this test shows evidence of a further decrease of the measured value of $K_{\text{I-init}}$. Otherwise, some indication of the displacement rate dependence of $K_{\text{I-init}}$ can be gleaned by plotting the measured values of $K_{\text{I-init}}$ as a function of the applied displacement rate to establish whether the curve appears to be asymptotic to the K_{QSCC} value, as illustrated in Figure 13.

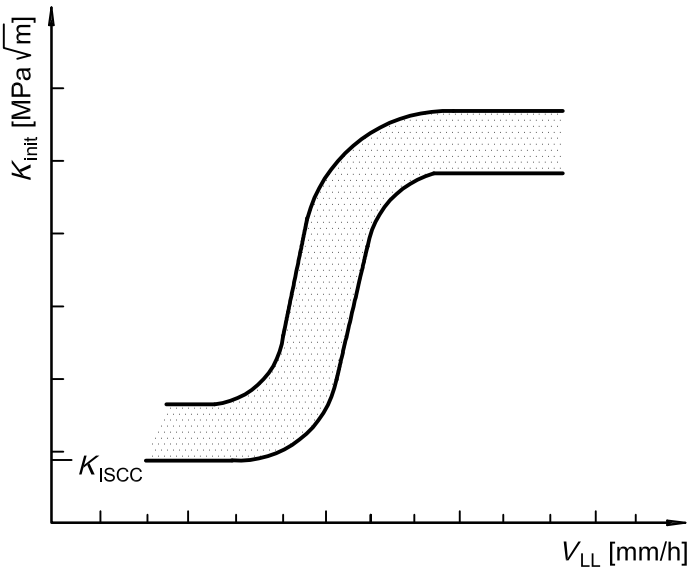


Figure 13 — Initiation value of stress intensity factor as a function of the applied displacement rate

7.6.3 Validation of test results

The result of the test series is valid, i.e. $K_{QSCC} = K_{ISCC}$, unless

a) the difference between any two of these last three measurements exceeds 2,5 % W;

b) the difference between the maximum and minimum crack lengths exceeds 5 % W;

c) any part of the fatigue crack surface lies in a plane the slope of which exceeds an angle of 10° from the plane of the notch:

d) the fatigue crack is not in one plane, i.e. effects of multi-nucleation are present;

e) the factor $2{,}5\left(\frac{K_{I\text{-init}}}{R_{p0,2}}\right)^2$ is greater than the thickness of the specimen and/or the crack length;

f) there is uncertainty over the fatigue crack length.

7.7 Determination of crack velocity

7.7.1 Tests performed in 7.6 which yielded values of $K_{I\text{-init}}$ in agreement of K_{ISCC} (or K_{QSCC}) can be used for determining the velocity of the environmental crack growth either as average data or as a function of the stress intensity factor K, following the procedure given in Annex B. To determine these data, the specimens shall be broken and the fracture surfaces examined by microscopic means.

7.7.2 The final crack front shall be measured, if possible to the nearest 0,5 % W, at both edges and at the following three positions:

0,25 B; 0,50 B and 0,75 B.

The average of these five measurements shall be used as the effective final crack length, a_f.

7.7.3 The average crack velocity $\Delta a/\Delta t$ is obtained by dividing the difference between the final and the initial crack length, $a_f - a_0$, by the time elapsed between crack initiation and termination of the test.

8 Test report

The test report shall contain at least the following information.

a) Full description of the test material from which the specimens were taken, including composition, structural condition and mechanical properties, type of product and section thickness. K_{Ic} (or K_Q if the validity criteria are not obeyed) if determined.

b) Description of the test machine and equipment used to measure crack length and the precision with which crack length measurements were made.

c) Descriptions of the environmental chamber and all equipment used for environmental monitoring control.

d) The initial solution composition, pH, degree of aeration (or concentration of other relevant gases), flow conditions, temperature and electrode potential reported, where monitored. Specification of flow rate shall be in terms of approximate linear rate past specimen if determined by the recirculation rate.

NOTE The reference electrode used should be indicated; the potential should be reported and referred to an appropriate standard electrode (e.g. the standard hydrogen electrode or saturated calomel electrode at 25 °C). Variations in these parameters during testing should be recorded.

e) The starting procedure for the test.

f) Details of transients in the environment or in the loading (including test interruptions) during testing, noting the nature and duration and, where applicable, the associated crack lengths.

g) For each specimen:

   1) specimen type and loading method;

   2) thickness, B, in millimetres (and B_n if side-grooved);

   3) width, W, in millimetres;

   4) fatigue cracking

      i) the fatigue stress intensity factor, K_f, during the propagation of the final portion of the crack;

      ii) the fatigue load ratio, R;

      iii) the temperature and environment during precracking;

   5) the length of the fatigue pre-crack, a;

   6) the initial stress intensity, K_{Ii};

   7) the initial time of exposure to the environment and the total time of testing;

   8) whether stable crack extension occurred;

   9) crack plane and propagation direction, identified as shown in Figure 10.

h) Range of displacement rates used.

i) K_{ISCC} (or K_{QSCC} if the validity criteria are not obeyed), stating at which displacement rate determined and criteria used.

j) Crack growth data (average values or as a function of stress intensity) where available.

Annex A
(informative)

Determination of a suitable displacement rate for determining K_{ISCC} from constant displacement rate tests

A.1 General

This procedure is based on the assumption that the displacement rate, $(dq/dt)_{SCC}$, at which a test in the corrosive environment has to be performed in order to evaluate the K_{ISCC} value of a system, can be estimated from the ratio of measured crack growth velocity in an inert environment, $(da/dt)_{inert}$, and the crack growth velocity in the plateau region for environmentally induced cracking, $(da/dt)_{SCC}$, by

$$(dq/dt)_{SCC} < \frac{(da/dt)_{SCC}}{(da/dt)_{inert}}(dq/dt)_{inert}$$

The crack growth velocity for environmentally induced cracking, $(da/dt)_{SCC}$, can be obtained within reasonable time from a test which avoids long incubation periods by applying high stress intensity levels. This can be constant displacement tests using compact tension, DCB or WOL specimens in accordance with ISO 7539-6, which are terminated after sufficient crack extension has been observed to roughly estimate $(da/dt)_{SCC}$. Step loading tests are equally well suited for this purpose. It has been found that average crack velocity data which are determined from slow strain rate tests on smooth specimens in accordance with ISO 7539-7, under the same environmental conditions, can be favourably used as a lower bound value.

A.2 Procedure

A.2.1 A specimen of similar size and shape to the specimens used for the subsequent SCC tests is tested in air (or in an inert environment) at a loading rate that results in a load-point displacement rate, $(dq/dt)_{inert}$, between 0,1 W/min and 0,1 W/h. The load-displacement behaviour of the specimen and the time elapsed since the start of the test are measured and recorded. The test is continued until a crack extension of at least 0,1 W is measured.

A.2.2 The extent of crack growth is marked by either heat tinting or fatigue cracking.

A.2.3 The specimen is broken to reveal the fracture surface and the initial and final crack lengths, a_0 and a_f, are measured at both edges and at the following three positions:

0,25 B, 0,50 B and 0,75 B.

The averages of these five measurements are used as the effective crack lengths a_0 and a_f.

A.2.4 The average inert crack growth velocity $(\Delta a/\Delta t)_{inert}$ is determined by dividing the difference between the final and the initial crack length, $a_f - a_0$, by the time elapsed between the crack initiation and the time of test termination

A.2.5 The crack growth velocity, $(da/dt)_{SCC}$, for stress corrosion cracking is determined from any of the methods mentioned in Annex C.

A.2.6 The appropriate displacement rate for the rising displacement test for determining K_{ISCC} is obtained by

$$(dq/dt)_{SCC} = 0{,}5\frac{(da/dt)_{SCC}}{(\Delta a/\Delta t)_{inert}}(dq/dt)_{inert}$$

Annex B
(informative)

Determination of crack growth velocity

The crack growth velocity, da/dt, is calculated from the crack length versus elapsed time data (a versus t). If the data are noisy or there are unrealistic outliers, it may be advisable to fit the a vs t curve with a polynomial prior to calculation of crack growth rates, otherwise apparent oscillations in crack growth may arise. Care must be taken to ensure that real variations in a vs t are not obscured by the fitting procedure.

One recommended method is the Incremental Polynomial Method as described in ISO 11782-2:1997 for the evaluation of fatigue crack growth rates (da/dN). This method involves fitting a second-order polynomial (parabola) to sets of ($2n$ + 1) successive data points, where n is usually 1, 2, 3 or 4.

The form of the equation for the local fit is as follows:

$$\hat{a} = b_0 + b_1 \frac{T_\mathrm{i} - C_1}{C_2} + b_2 \left(\frac{T_\mathrm{i} - C_1}{C_2} \right)^2$$

provided that:

$$-1 \leqslant \frac{T_\mathrm{i} - C_1}{C_2} \leqslant +1$$

T_i is the time elapsed, b_0, b_1, and b_2 are the regression parameters that are determined by the least squares method over the range $a_{\mathrm{i}-n} \leqslant a \leqslant a_{\mathrm{i}+n}$. The value $\hat{a}_\mathrm{i}$ is the fitted value of crack length at T_i.

The parameters $C_1 = ½(T_{\mathrm{i}-n} + T_{\mathrm{i}+n})$ and $C_2 = ½(T_{\mathrm{i}+n} - T_{\mathrm{i}-n})$ are used to scale the input data. The crack growth velocity at T_i is obtained from the derivative of the above parabola, which is given by

$$(\mathrm{d}a/\mathrm{d}t)\hat{a}_i = \frac{b_1}{C_2} + 2b_2 \frac{T_\mathrm{i} - C_1}{{C_2}^2}$$

The value of K_I associated with the da/dt value is computed using the fitted crack length, $\hat{a}_\mathrm{i}$, corresponding to T_i.

Annex C
(informative)

Information on indirect methods for measuring crack length

C.1 Electrical resistance measurement methods

C.1.1 DC potential drop method

The specimen is electrically insulated and a constant current passed through it, across the crack plane. Typically the current density used on a ferritic steel specimen will be of the order of 10^4 to 10^5 A/m^2 on the net section, but higher current densities may be required on metals of lower resistivity. The potential drop between two points on either side of the crack plane is monitored and a calibration, derived either experimentally, analytically or numerically, used to relate this to crack length. In the latter cases, experimental verification is desirable.

The method is well established and proprietary equipment with the necessary stability and reliability is readily available. It may be not well suited to large specimens or those of low resistivity because of the very high currents that may be required. A derivative of the method exists however which can be used in these circumstances. This involves monitoring the length of the crack in a brittle metal foil attached to the specimen, the length of crack in the foil being taken to be equal to the surface length of the crack in the underlying specimen.

Potential sources of error with the general method are as follows:

a) thermal resistivity changes;

b) thermal electromagnetic forces (emfs).

A comparator technique may be used to overcome a). The potential drop across the crack is divided by the potential drop either measured between points on a second specimen similar to the first, electrically in series with it and physically close to it, but not cyclically loaded, or between a second pair of probes on the specimen under test. The latter, though experimentally easier, requires care in calibration as both the measured potentials may vary with crack length.

Thermal emfs can be eliminated by using voltage probes of similar material to the specimen. When junctions between two dissimilar metals are inevitable, they should be embedded within a relatively large thermal mass. A split, hollowed-out cube of aluminium of 50 mm side is suitable

The method provides a measure of the average crack length across the specimen and is well suited to automatic data collection and machine control.

C.1.2 AC potential drop methods

Potential drop methods fall into two categories: low- and high-frequency systems.

The low-frequency systems (typically operating in the range 10 Hz to 100 Hz) are essentially developments of the DC method. The use of phase-sensitive detection systems enables a high signal:noise ratio to be obtained and thus the sensitivity is enhanced. Thermal emfs also cease to be a problem. However, calibrations are required as with the DC method.

High-frequency systems (5 kHz to 8 kHz) make use of the localization of current flow to the “skin” of the specimen that occurs at these frequencies. This minimizes the current requirements and leads to a linear relationship between voltage and crack length which is independent of specimen size. The method is thus particularly suitable for measuring cracks in large specimens.

The electronics of AC systems are relatively complex and there may be difficulty in achieving the required long-term stability. Depending on the particular characteristics of the system, great care may be necessary to avoid spurious signals due to pick-up. Thus, the physical loop formed between the probe wires and the specimen surface should be kept as small as possible and the probe wires should be twisted together or miniature screened/shielded cable used. Wires should be screened to prevent their movement and the probe and field current wires well separated.

Incorporating the higher-frequency methods into automatic monitoring systems is particularly convenient due to the linear voltage to crack length relationship obtained.

C.2 Compliance methods

The two most popular methods are as follows:

a) measurement of the displacement per unit load across the notch;

b) measurement of the “back face strain” per unit load.

In a) a suitable transducer, e.g. clip-on extensometer, LVDT (Linear Variable Displacement Transducer) or ring dynamometer is mounted across the notch mouth (in the case of SENB3, SENB4 and CT specimens) or notch centre (in the case of CCT specimens). The displacement per unit load is determined and related by calibration to the crack length. If a second specimen is used, it should be placed under identical environmental conditions as the test specimen. This will ensure similar transient thermal, etc. behaviour. Thermal effects can also be minimized by using a reversing polarity technique.

The “back face strain” (BFS) method is similar in principle. In this instance, the transducer is a strain gauge bridge located on the “back face” of the specimen, i.e. that opposite the notch. This method has been particularly successful with CT specimens and similar sensitivity would be expected with SENB4. It is not suitable for SENB3 due to the location of a loading point opposite the notch, nor for CCT as BFS in this geometry is not very sensitive to crack length. BFS may be preferred to other compliance methods at high frequencies due to the practical absence of inertial effects. Displacement transducers and strain gauges should be kept out of contact with the solution to preserve their integrity and to avoid galvanic effects.

The methods provide a measure of the average crack length and are well suited to automated data collection and machine control.

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