
**Corrosion of aluminium alloys —
Determination of resistance to stress
corrosion cracking**

*Corrosion des alliages d'aluminium — Détermination de la résistance à
la corrosion fissurante sous contrainte*



Reference number
ISO 9591:2004(E)

© ISO 2004

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

© ISO 2004

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

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

Published in Switzerland

Contents

Page

1	Scope	1
2	Normative references	1
3	Terms and definitions	1
4	Principle	2
5	Apparatus	2
6	Sampling	4
7	Specimens	6
8	Test environment	7
9	Stress considerations	8
10	Procedure	8
11	Assessment of results	9
12	Test report	9
	Annex A (normative) Grain orientation examination	10
	Bibliography	11

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 9591 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

This second edition cancels and replaces the first edition (ISO 9591:1992), which has been technically revised.

Corrosion of aluminium alloys — Determination of resistance to stress corrosion cracking

1 Scope

1.1 This International Standard specifies a method for the determination of resistance to stress corrosion cracking (SCC) of aluminium alloys.

1.2 This International Standard covers the method of sampling, the types of specimens, the loading procedure, the type of environment and the interpretation of results.

1.3 This International Standard is aimed at determining resistance to SCC as a function of the chemical composition, the method of manufacture and heat treatment of aluminium alloys.

1.4 This International Standard applies to cast and wrought aluminium alloys in the form of castings, semi-finished products, parts and weldments.

1.5 Since most natural and many artificial environments contain chlorides, this International Standard can be used to compare the performance of products employed under marine atmospheres and in environments containing chlorides, providing that the failure mechanism is not changed. However, the results of this test should not be considered as an absolute criterion for the quality of alloys.

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 7539-2:1989, *Corrosion of metals and alloys — Stress corrosion testing — Part 2: Preparation and use of bent-beam specimens*

ISO 7539-3:1989, *Corrosion of metals and alloys — Stress corrosion testing — Part 3: Preparation and use of U-bend specimens*

ISO 7539-4:1989, *Corrosion of metals and alloys — Stress corrosion testing — Part 4: Preparation and use of uniaxially loaded tension specimens*

ISO 7539-5:1989, *Corrosion of metals and alloys — Stress corrosion testing — Part 5: Preparation and use of C-ring specimens*

ISO 7539-6:2003, *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*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 7539-1 apply.

4 Principle

4.1 This International Standard specifies two methods of loading:

- under constant total strain;
- under constant load.

It does not cover slow strain rate test methods and determination of maximum admissible stress by the method of permanent deformation for aluminium alloys, although an effort is now being made to apply such methods to these alloys (see ISO 7539-7).

4.2 This International Standard specifies two methods of immersion in the solution:

- alternate immersion;
- continuous immersion (subject to agreement between the interested parties).

NOTE 1 In alternate immersion experiments, the exposure of the wetted surface to the atmosphere and the subsequent drying out create an aggressive salt environment on the metal surface and enhanced transport of oxygen prior to subsequent immersion. The rate of drying will depend on the nature and thickness of the salts on the metal surface and may not be the same for different alloys.

NOTE 2 The wetting and drying cycle mimics, to some extent, the wetting and drying in marine atmospheres (although salts on the metal surface may redissolve on subsequent immersion in the laboratory tests).

4.3 The evaluation criteria for corrosion cracking of alloys are:

- σ_{SCC} : the threshold stress, which is the maximum stress under which no failure of the samples occurs during the fixed period of the test;
- τ_{SCC} : the time of failure, which is the moment of the appearance of the first visible crack (or under magnification up to $\times 30$) for specimens under constant strain.

4.4 The selection of the method of loading, the value of stresses, corrosive environment and criteria of evaluation can be the subject of an agreement between the interested parties and should be defined by the test programme.

5 Apparatus

5.1 Loading apparatus

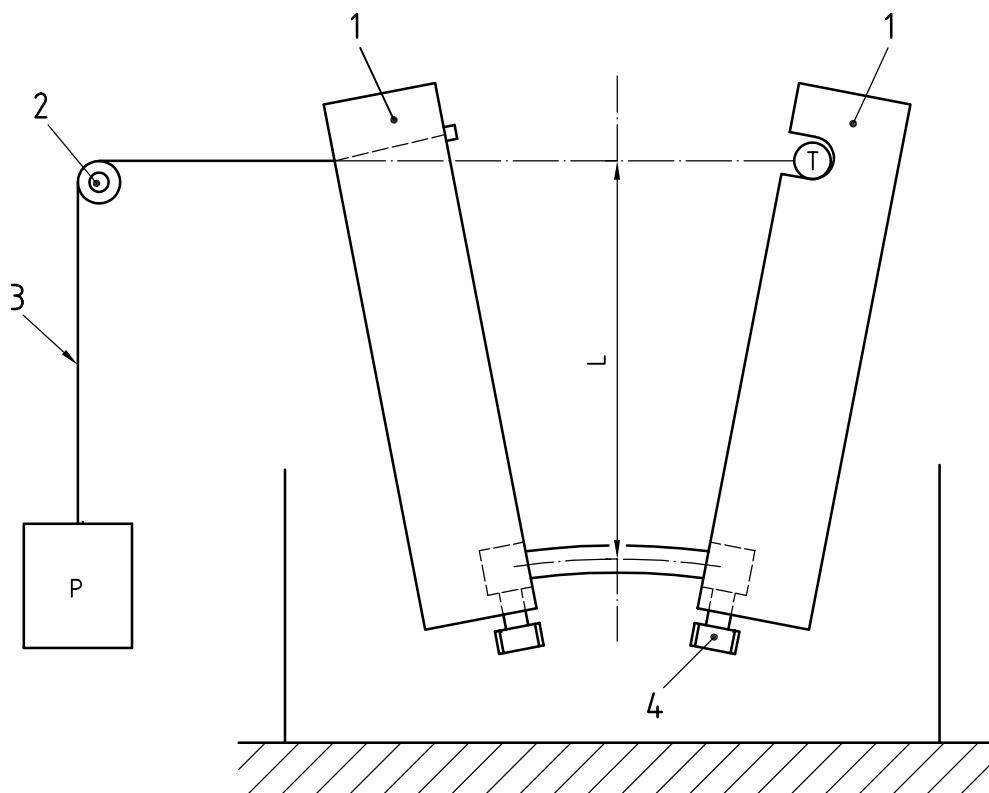
Tensile stresses in the specimens are produced with yokes, stressing screws, springs, lever devices and special testing machines, e.g., testing devices for constant bending (Figure 1) and for constant load (Figure 2). The latter figure is an example of a device for multiple specimen testing which can be a considerable advantage.

5.2 Construction materials

If in contact with the salt solution, the materials shall not be affected by the corrodent to such an extent that they can cause contamination of the solution and change its corrosiveness.

NOTE 1 Use of inert plastics or glass is recommended where feasible.

NOTE 2 Metallic components that are in contact with the solution should be made from corrosion resistant materials such as those recommended for marine environments. These materials should not be affected by the solution to the extent that they can cause contamination of the solution. In addition, metallic components may be protected by coating materials that do not cause contamination of the solution or change its corrosiveness.

**Key**

- 1 perspex
- 2 pulley
- 3 nylon cord
- 4 nylon bolt

Figure 1 — Constant load bending device**5.3 Specimen holders**

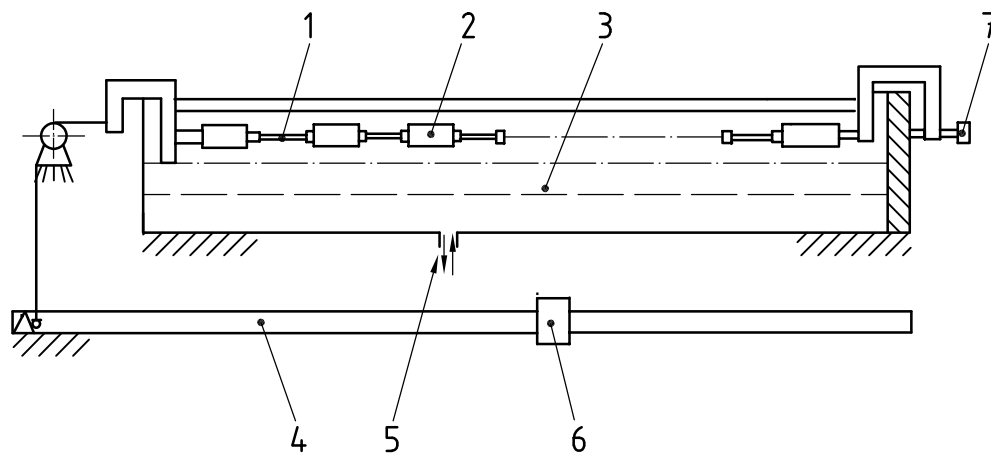
The specimen holders shall be designed to electrically insulate the specimens from each other and from any bare metal parts. When this is not possible, as in the case of certain stressing bolts or jigs, the bare metal contacting the specimen shall be isolated from the corrodent by a suitable coating. Protective coatings shall be of a type that will not leach inhibiting or accelerating ions or protective oils or leave any residue, e.g. vapour, on the non-coated portions of the specimen. In particular, coatings containing chromates shall be avoided. It is recommended that all samples be degreased after coating.

5.4 Apparatus for alternate immersion in solutions

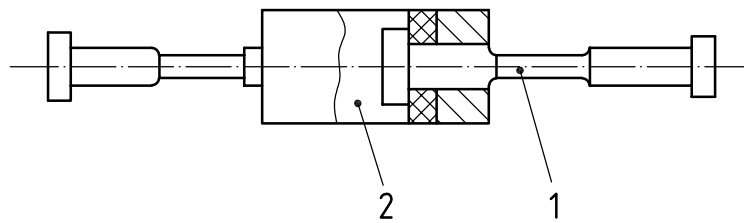
5.4.1 The temperature controller shall be capable of increasing the temperature of the surface of the specimen from 0 °C to 100 °C at a controlled rate. This is achieved by heating the solution. Above 10 °C, the average rate of temperature change of the specimen shall be controlled to within $\pm 30\%$ of the desired value, where the average is calculated over a temperature range of 10 °C. Guidelines for calculating the temperature of the specimen relative to the temperature of the solution are given in Clause 7.

5.4.2 Any suitable mechanism may be used to accomplish the immersion portion of the cycle provided that:

- it achieves the specified rate of immersion and removal;
- the apparatus is constructed of suitable inert materials.



a) Multiple test assembly



b) Chains of specimens connected by loading links

Key

- 1 specimen
- 2 loading link
- 3 environment
- 4 lever system
- 5 connexions for solution sleeve
- 6 mobile load
- 7 loading screw

Figure 2 — Illustrations of the multiple specimen testing device for the constant uniaxial stress corrosion cracking test

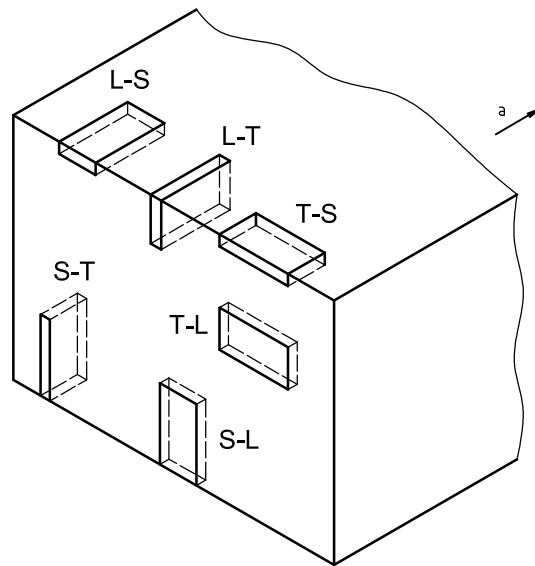
The usual methods of alternate immersion are:

- a) specimens are placed on a movable rack that is periodically lowered into a stationary tank containing the solution;
- b) specimens are placed on a corrosion wheel arrangement which rotates every 10 min through 60° and thereby passes the specimens through a stationary tank of solution;
- c) specimens are placed in a stationary tray open to the atmosphere and the solution is moved by air pressure, by a non-metallic pump, or by gravity drain from the reservoir to the tray.

5.4.3 The rate of immersion and removal of the specimens from the solution should be as rapid as possible without jarring them. For the purposes of standardization, an arbitrary limit shall be adopted such that no more than 2 min elapse for the time to achieve full immersion or emersion from the solution.

6 Sampling

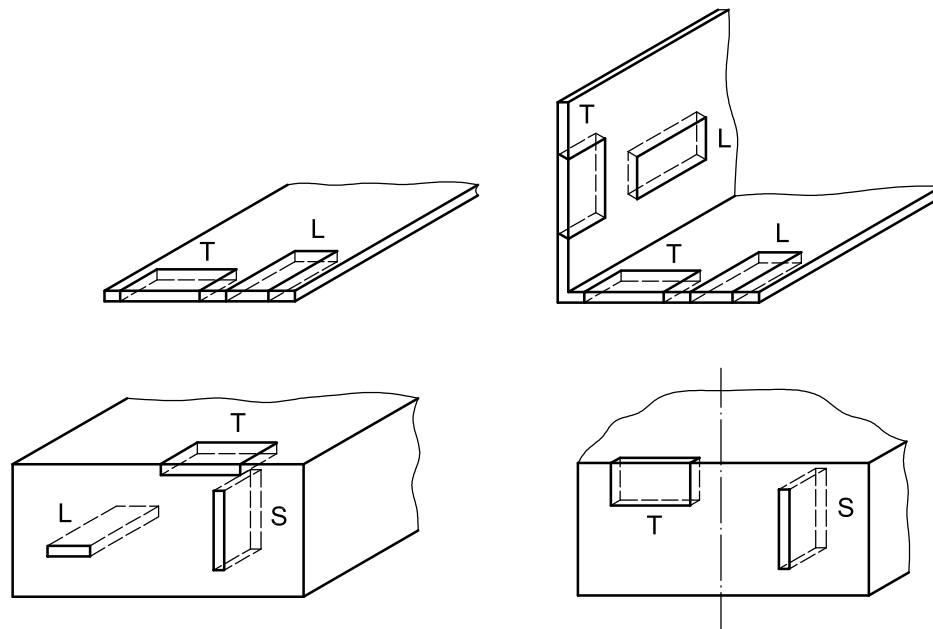
6.1 In general, this International Standard specifies three specimen orientations for thick products and two for thin products. The orientation diagram is given in Figure 3. In Figure 3 a), the first direction refers to the specimen axis and the second direction refers to the direction of crack growth.



Key

- a Working direction
- L—T = longitudinal-long transverse direction
- L—S = longitudinal-short transverse direction
- T—L = long transverse-longitudinal direction
- T—S = long transverse-short transverse direction
- S—L = short transverse-longitudinal direction
- S—T = short transverse-long transverse direction

a) General procedure



b) Recommended procedure

Key

- L = longitudinal direction
- T = long transverse direction
- S = short transverse direction

Figure 3 — Specimen orientation

6.2 Unless otherwise specified, tests shall be performed in the short transverse direction (S) for thick products and in the long transverse direction (T) for thin products. In rolled or extruded sections that are approximately round or square, the samples shall be oriented in the transverse (diametrical) direction. In the case of forgings and, more generally, when the structure of the test pieces is not obvious, it is recommended that the grain direction be determined by macroetching or by metallographic examination, in order to select specimens in the most susceptible (short transverse or long transverse) directions (see Annex A).

6.3 The number of specimens to be submitted for testing and which stress levels are to be used shall be agreed upon between the interested parties. For each stress level no fewer than three adjacent specimens shall be tested under stress. If desired, an additional specimen may be exposed unstressed. The number of stress levels will depend on the objective of the testing: production quality assurance or alloy/process development.

7 Specimens

7.1 Type and sizes

Specimens as defined by ISO 7539-2, ISO 7539-3, ISO 7539-4, ISO 7539-5 and ISO 7539-6 can be used.

- a) Tension specimens, C-ring specimens or bend specimens can be taken from thick products, e.g. from plates or forgings.
- b) Tension specimens, bent beam specimens or U-bend specimens can be taken from thin products, e.g., from sheets.
- c) When specimens are taken from weldments where the weld is perpendicular to the stress axis, the weld shall be placed in the middle of the specimen.
- d) The comparison of different alloys and tempers shall be conducted on specimens of the same type and size. Where possible, specimens shall be heat treated before final machining, otherwise consideration shall be given to the removal of oxidation products from the surface (see 7.2.3).

7.2 Surface preparation

7.2.1 Specimens with surface defects (of mechanical or metallurgical origin) shall be discarded.

7.2.2 The surface quality of a specimen shall comply with the following:

- without mechanical machining, in the as-supplied condition;
- with mechanical machining: an arithmetical mean deviation of the profile, Ra , $\leq 1 \mu\text{m}$ is recommended, unless it is required to simulate an as-manufactured surface condition;
- the surface condition of welded specimens shall be agreed upon between the interested parties.

NOTE 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. In addition, near-surface residual stresses can be introduced during machining and surface grinding and these processes should be undertaken in such a way as to minimize the generation of residual stress. In some cases, relief of surface residual stress may be undertaken provided that this does not lead to a change in microstructure. Measurement of residual stress is desirable.

7.2.3 Test specimens shall be degreased using an organic solvent (e.g. acetone) then etched in a 5 % to 10 % sodium hydroxide solution at a temperature of 50 °C to 60 °C for 0,5 min to 2 min, rinsed in running water, cleaned in a 30 % solution of nitric acid, rinsed in running water, rinsed in distilled water at 70 °C to 90 °C and dried.

7.2.4 After degreasing, it is possible to use the following etching conditions as an alternative to the procedure described in 7.2.3.

1 min at 95 °C in a solution of:

nitric acid (63 % mass fraction)	50 ml/l
hydrofluoric acid (60 % mass fraction)	5 ml/l
water	945 ml

followed by rinsing in running and distilled water (as in 7.2.3) then drying.

7.2.5 Specimens with mechanically machined surfaces need not be etched.

7.3 Specimen identification

Specimens shall be identified by a suitable method (see ISO 7539-1) and the markings protected against corrosion.

7.4 Precautions

After the cleaning procedure, care shall be taken to avoid handling the stressed region of the specimen.

8 Test environment

8.1 The test environment shall be relevant to the intended application. The following environments are recommended. Other solutions may be used upon agreement between interested parties.

8.2 Analytical grade chemicals are recommended for the preparation of the corrosive solutions.

8.3 The solution shall be prepared using distilled or deionized water. The minimum resistivity shall be $10^5 \Omega \cdot \text{cm}$.

8.4 For alternate immersions, an aqueous solution of sodium chloride with a mass fraction of $(3,5 \pm 0,1) \%$ shall be used. The pH of this solution shall be between 6,4 and 7,2, following adjustment, if necessary (see 8.6).

8.5 For continuous immersion, an aqueous solution containing mass fractions of $(2,0 \pm 0,1) \%$ sodium chloride and of $(0,5 \pm 0,05) \%$ sodium chromate (Na_2CrO_4) shall be used.

The pH of this solution shall be adjusted to within $3,0 \pm 0,2$ using hydrochloric acid.

The pH shall be checked regularly during the test and adjusted, if necessary (see 8.6).

8.6 Only diluted hydrochloric acid or sodium hydroxide solution shall be used to adjust the pH of test solutions.

8.7 The temperature and humidity of the laboratory shall be controlled for the purpose of inducing a drying rate sufficient to dry out the specimens prior to re-immersion. The following conditions are commonly used:

- a) temperature 18 °C to 25 °C; relative humidity 40 % to 70 %
- b) temperature 26 °C to 28 °C; relative humidity 40 % to 50 %.

Test conditions a) are used often when the samples are exposed to the laboratory atmosphere in an open chamber whilst, b) represents more tightly controlled laboratory conditions or use of a special chamber.

NOTE An indication of the rate of drying on the surface can be made by observing test materials or by using a bimetallic corrosion probe. Such measurements may be useful when initiating a new or renovated facility. Such measurements are not usually made on a continuing basis.

8.8 Before the freshly prepared solution is used, its temperature shall be within 3 °C of the specified air temperature. Thereafter, no control of the solution temperature is required. Instead, the ambient temperature is controlled and the solution is allowed to reach temperature equilibrium.

8.9 The minimum ratio between the volume of test solution and the exposed area of specimens (including any uncoated accessories made from aluminium alloys) shall be 35 ml/cm² for alloys exposed under alternate immersion conditions, and 10 ml/cm² for alloys exposed under continuous immersion conditions in sodium chloride/sodium chromate solution.

8.10 Evaporation losses shall be made up by daily additions of water of the required purity (see 8.3).

8.11 Fresh solutions shall be prepared weekly for alloys containing more than 0,5 % copper and every 15 days for other alloys. At the same time, the portions of the apparatus in contact with the solution shall be cleaned by flushing with water.

9 Stress considerations

9.1 The loads shall be such that the stresses are elastic. For specimens of the type described in ISO 7539-2, ISO 7539-3, ISO 7539-4, ISO 7539-5 and ISO 7539-6, the relevant standard shall be consulted for calculation of the elastic stresses.

In the case of calculation of circumferential stresses in the C-ring specimens, the best results can be obtained when change of diameter (ΔD) is measured by a digital linear indicator with an accuracy of not less than 0,01 mm. Electrical gauges shall be used when it is necessary to take into account circumferential and transverse stresses, while in this case nominal length of a gauge shall be not more than 5 mm.

9.2 The maximum tensile stress in bend specimens in the form shown in Figure 1 is given by the following equations.

Test on flat specimen:

$$\sigma = 6 \times PL/Be^2 \times 10^{-6}$$

Test on plain cylindrical specimen:

$$\sigma = 10,2 \times PL/d^3 \times 10^{-6}$$

where

- σ is the stress in megapascals;
- P is the applied load in newtons;
- B is the width of specimen in metres;
- L is the length of the lever arm in metres;
- e is the thickness of specimen in metres;
- d is the diameter of calibrated section of specimen in metres.

10 Procedure

10.1 The specimens are exposed to the test environment as soon as possible after stressing.

10.2 During alternate immersion, the specimens are totally immersed in the sodium chloride solution for 10 min of each hour and then removed from the solution and allowed to dry for 50 min.

A mild circulation of air is recommended and stagnant air conditions shall be avoided.

Drying by a forced air blast on the specimens is not recommended because of the difficulty in maintaining uniform drying of large groups of specimens.

The tests are usually carried out without interruption during a given exposure time.

10.3 The test duration depends upon the composition and heat treatment of the alloy, the specimen size, the environment and the method of stressing. Usually, test durations of between 10 days and 90 days are used.

When the purpose of the test is to control the quality of production lots or to determine the characteristics laid down in the standard documents concerning alloys, the test duration shall be as required in pertinent specifications or shall form the subject of an agreement between the interested parties.

10.4 Specimens of dissimilar alloy composition shall not be tested simultaneously in the same salt solution. In particular, specimens of materials belonging to different alloy series (2000, 5000, etc.), shall not be tested together.

10.5 After exposure, unfailed specimens shall be unloaded, rinsed with water, and then cleaned in concentrated nitric acid (60 % to 70 %) at room temperature to remove corrosion products. They shall then be rinsed with water and dried prior to more detailed examination.

11 Assessment of results

11.1 For specimens that do not fail within the specified exposure period, examination of a cross-section by optical metallography may be used to distinguish between stress corrosion, intergranular corrosion and transgranular pits. A specimen that reveals intergranular cracks deeper than transgranular pits shall be considered as an SCC failure. Intergranular corrosion not deeper than transgranular pits shall not be considered as an SCC failure.

11.2 For specimens that do fail, examination of the fracture surface by scanning electron microscopy (SEM) allows brittle intergranular failure due to stress corrosion to be distinguished from transgranular ductile failure due to mechanical overload. Both type of failures are present when SCC is the origin of failures for specimens under constant load.

12 Test report

The test report shall contain at least the following information:

- a) chemical composition or designation of the alloy;
- b) product, temper and section thickness of material tested;
- c) location and grain orientation with respect to the original material (see Figure 3);
- d) type of specimen and dimensions;
- e) surface condition of the specimens;
- f) level of stress and method of loading;
- g) chemical composition of the corrosive solution;
- h) immersion method (continuous immersion or alternate immersion);
- i) environmental exposure conditions (including pH, humidity and temperature);
- j) results of any metallographic examinations;
- k) number of replicate tests.

Annex A (normative)

Grain orientation examination

A.1 Macroetching

Conduct etching on a machined surface.

Immerse the specimen for about 1 min at room temperature in a solution composed of:

- 1 volume hydrochloric acid of mass fraction 38 %, $\rho = 1,19$ g/ml;
- 1 volume of nitric acid of mass fraction 60 %, $\rho = 1,38$ g/ml;
- 2 % (volume fraction) of hydrofluoric acid of mass fraction 40 %, $\rho = 1,14$ g/ml;

made up to 3 volumes with water.

Visually inspect with the naked eye or at low magnifications.

A.2 Fluoboric etching

Conduct etching on a carefully polished surface, preferably after electrolytic polishing.

Anodic oxidation: 20 mA/cm² in an aqueous solution of 2,5 % (volume fraction) fluoboric acid for 1 min or 2 min at room temperature.

Examine using a microscope in polarized light.

Bibliography

- [1] ISO 7539-8:2000, *Corrosion of metals and alloys — Stress corrosion testing — Part 8: Preparation and use of specimens to evaluate weldments*
- [2] ISO 7539-9:2003, *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*
- [3] SINIAVSKI, V.S., VALKOV, V.D. and KALININ, V.D. *Corrosion and Protection of Aluminium Alloys*, Moscow Publishing House, Metallurgia (1986)
- [4] NF A 05-301, *Alliages d'aluminium essai de corrosion sous contrainte des produits épais*
- [5] GOST 9.019-74, *Aluminium and Aluminium Alloys — The Methods of Acceleration Tests on Stress-Corrosion Cracking*
- [6] ASTM G44-99, *Standard Practice for Exposure of Metals and Alloys by Alternate Immersion in Neutral 3.5 % Sodium Chloride Solution*
- [7] ASTM G47-98, *Standard Test Method for Determining Susceptibility to Stress-Corrosion Cracking of 2XXX and 7XXX Aluminum Alloy Products*

.....

ICS 77.060

Price based on 11 pages