INTERNATIONAL STANDARD

ISO 20728

First edition
2018-09

Corrosion of metal and alloys — Determination of resistance of magnesium alloys to stress corrosion cracking

Corrosion des métaux et alliages — Détermination de la résistance des alliages de magnésium à la fissuration par corrosion sous contrainte

Reference number
ISO 20728:2018(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword ..... **iv**

1 Scope **1**

2 Normative references **1**

3 Terms and definitions **1**

4 General principles **2**

5 Apparatus and materials **2**
5.1 Loading apparatus 2
5.2 Construction materials for the test set-up 2
5.3 Specimen holders 2
5.4 Apparatus for alternate immersion in solutions 3

6 Sampling **3**

7 Specimens **5**
7.1 Type and sizes 5
7.2 Surface preparation 5
7.3 Specimen identification 6
7.4 Precautions 6

8 Test environments **6**

9 Loading **7**

10 Procedure **7**

11 Assessment of results **8**

12 Expression of results **9**

13 Test report **9**

Annex A (informative) **Assessment of grain size and orientation** **11**

Bibliography **12**

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Corrosion of metal and alloys — Determination of resistance of magnesium alloys to stress corrosion cracking

WARNING — This document calls for the use of substances and/or procedures that can be injurious to health if adequate safety measures are not taken. This document does not address any health hazards, safety or environmental matters associated with its use. It is the responsibility of the user of this document to establish appropriate health, safety and environmentally acceptable practices.

1 Scope

This document specifies a method for the determination of resistance to stress corrosion cracking (SCC) of magnesium alloys intended for use in structural applications (such as magnesium front end, gearbox and clutch housing units, steering column parts, shift actuators, valve covers and housings, brackets and intake manifold blades, electronic devices, power tools and medical equipment). The method allows determination of the resistance to SCC as a function of the chemical composition, the method of manufacture and heat treatment of magnesium alloys.

The document is applicable to cast and wrought magnesium alloys in the form of castings, semi-finished products, parts and weldments and covers the method of sampling, the types of specimens, the loading procedure, the type of environment and the interpretation of results.

The document allows assessment of the relative performance of materials and products in environments containing chlorides or sulphates, provided that the failure mechanism is not changed, but does not qualify a material or product for service application.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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, *Corrosion of metals and alloys — Stress corrosion testing — Part 1: General guidance on testing procedures*

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

ISO 7539-7:2005, *Corrosion of metals and alloys — Stress corrosion testing — Part 7: Method for slow strain rate testing*

ISO 8044, *Corrosion of metals and alloys — Basic terms and definitions*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8044 and ISO 7539-1 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 General principles

4.1 Stress corrosion cracking of magnesium alloys is sensitive to microstructural orientation with respect to the stress axis. Accordingly, in testing SCC resistance, it is necessary to consider the manner in which the specimens are prepared from cast or wrought alloy so that orientation dependent SCC resistance can be assessed.

4.2 The corrosion of magnesium is associated with hydrogen evolution and generation of often very soluble corrosion products. For that reason, testing in stagnant conditions is preferred during continuous immersion as stirring can cause secondary effects, e.g. removal of corrosion product.

4.3 Two methods of immersion in the solution are proposed:

— alternate immersion;

— continuous immersion.

4.4 Tests may be conducted under constant load, constant strain or by the slow strain rate technique with evaluation criteria for stress corrosion cracking resistance appropriate to the chosen loading method.

4.5 The method of loading, the value of stresses, corrosive environment and criteria of evaluation should be agreed between the interested parties according to the purpose of the testing.

5 Apparatus and materials

5.1 Loading apparatus

Tensile stresses in the specimens are produced with yokes, stressing screws, springs, lever devices and special testing machines.

5.2 Construction materials for the test set-up

5.2.1 If in contact with the corrosive environment, the construction materials for the test set-up shall not be affected by the corrodent to such an extent that they can cause contamination of the solution and change its corrosiveness.

5.2.2 Use of inert plastics or glass is recommended for the corrosion cell where feasible.

5.2.3 Metallic components in contact with the solution shall be made from an appropriate corrosion resistant material, or protected with a suitable corrosion-resistant coating, sufficient to avoid galvanic coupling.

5.3 Specimen holders

5.3.1 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 holder. In particular, coatings containing chromates or releasing any other contaminants should be avoided. All samples holders should be degreased before and after coating.

5.3.2 The equipment required for slow strain rate testing is a device that permits a selection of strain rates while being powerful enough to cope with the loads generated. Strain rates that have been used most frequently in testing initially plain specimens are in the range 10^{-7} s^{-1} to 10^{-5} s^{-1}.

5.4 Apparatus for alternate immersion in solutions

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

a) it achieves the specified rate of immersion and removal, and

b) the apparatus is constructed of suitable inert materials.

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,

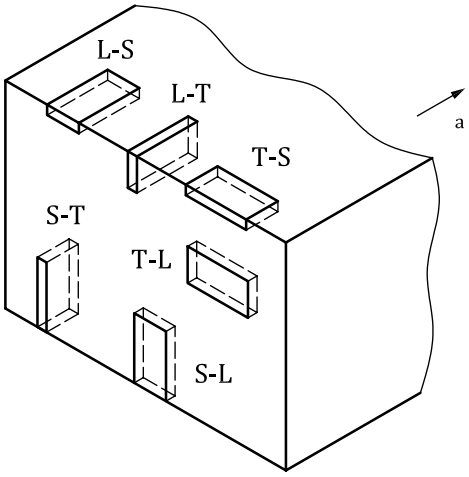
b) specimens are placed on a corrosion wheel arrangement which rotates every 10 min through 60° and thereby passes the specimens through a stationary tank of solution, and

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.

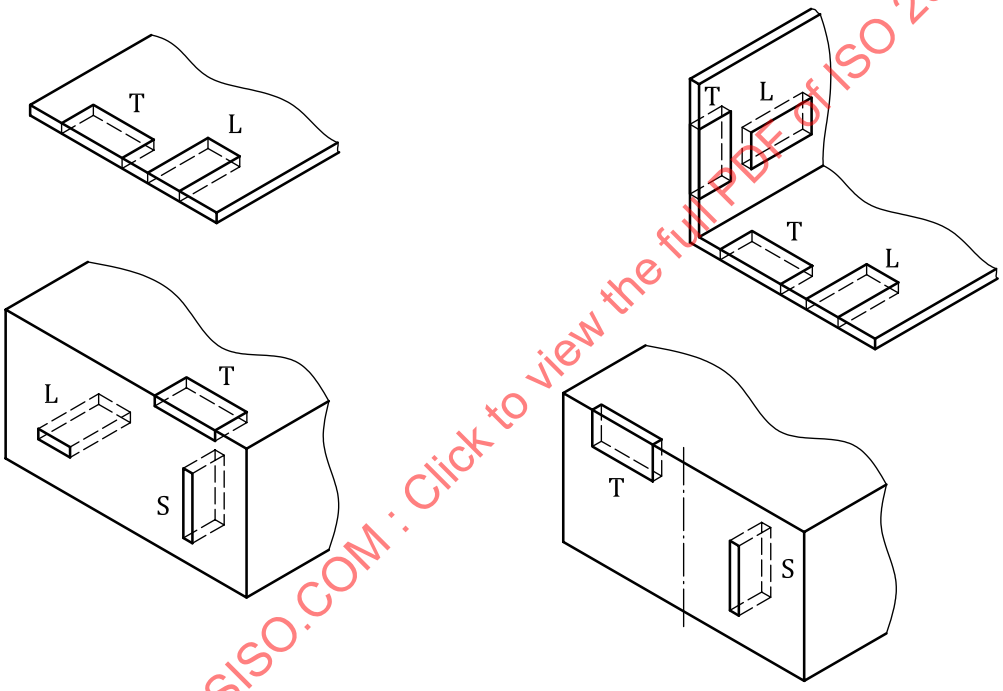
5.4.2 The rate of immersion and removal of the specimens from the solution shall be as rapid as possible without jarring them. For purposes of standardization, an arbitrary limit shall be adopted such that no more than 2 min elapses in the transfer from solution to air or vice versa.

6 Sampling

6.1 In general, this document specifies three specimen orientations for thick products and two for thin products. The orientation diagram is given in Figure 1. In Figure 1 a), the first direction refers to the stress axis and the second direction refers to the direction of crack growth.



a) General procedure



b) Recommended procedure

Key

Directions

L-T longitudinal-long transverse
L-S longitudinal-short transverse
T-L long transverse-longitudinal
T-S long transverse-short transverse
S-L short transverse-longitudinal
S-T short transverse-long transverse
L longitudinal direction
T long transverse direction
S short transverse direction
a Working direction.

Figure 1 — Specimen orientation

6.2 Unless otherwise specified, tests should be performed in the short transverse direction (S) for thick products and in the long transverse direction (T) for thin products.

For cast material, the preparation of samples from areas close to the casting surface should be avoided except in cases in which this area is intended to be studied. The local solidification rate while preparing the samples should be considered.

In rolled or extruded sections that are approximately round or square, the specimens should be oriented with the applied stress oriented in the transverse (diametrical) direction so that the crack path is in the rolling/extrusion direction. In any case, the sampling with respect to the orientation and/or to the rolling direction and/or by the texture should be reported.

In the case of forgings and, more generally, when the structure of the test pieces is not obvious, the grain direction should be determined by macro etching (see Annex A), or by metallographic examination, in order to select specimens in the most susceptible directions.

6.3 The number of specimens to be submitted to testing should be agreed between the interested parties. When testing at a single stress level only four or more specimens from the same location in the parent material shall be tested; or, if tests are being conducted at multiple stress levels three or more specimens per stress level are acceptable.

7 Specimens

7.1 Type and sizes

7.1.1 Specimens as defined by ISO 7539-2 to ISO 7539-6 can be used.

7.1.2 Tension specimens, C-ring specimens or bend specimens can be taken from thick products, for example from plates or forgings.

7.1.3 Tension specimens, bent beam specimens or U-bend specimens can be taken from thin products, for example from sheets.

7.1.4 Notched or pre-cracked specimens may be used when it is desired to measure threshold to cracking or crack growth rates. They may also be used to confine crack initiation and propagation to certain regions of the microstructure such as the heat affected zone of a weld. Notched or pre-cracked specimens may also be used to restrict load requirements, where bending, as opposed to tensile loading, can offer further advantages.

7.1.5 The comparison of different alloys and tempers should be conducted on specimens of the same type and size. Where possible, specimens should be heat treated before final machining, otherwise consideration must be given to the removal of oxidation products from the surface (see 7.2.4).

7.2 Surface preparation

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

— without mechanical machining: in the as-supplied condition;

— with mechanical machining: the arithmetical mean deviation of the profile (R_a) should be less than or equal to 1 µm, with a preferred value of 0,25 µm or finer, unless it is required to simulate an as-manufactured surface condition;

— the surface condition of welded specimens should be agreed between the interested parties.

7.2.2 Machining practices be designed to minimize the generation of residual stresses and changes to the near-surface metallurgical structure of the specimens.

7.2.3 Alternative surface treatments, for example pickling or etching, may be agreed between the interested parties. For cast specimens it should be specified whether the casting skin/scales should be removed prior to testing. For specimens taken from rolled products the surface should generally be cleaned of impurities by grinding, etching and/or polishing prior to testing.

7.2.4 Care shall be taken not to induce crack initiation by chemical and/or mechanical treatments.

7.2.5 Test specimens shall be degreased prior to testing. Suitable techniques comprise

— degreasing using an organic solvent, e.g. ethanol approx. 5 min in ultrasonic bath, and

— degreasing using alkaline solutions [15 min in NaOH at pH 12,5 and (87 ± 3) °C].

7.3 Specimen identification

Specimens shall be identified by a suitable method in accordance with ISO 7539-1 and the markings shall be protected against corrosion.

7.4 Precautions

After degreasing or pickling, the test region of the specimens shall not be touched prior to testing.

8 Test environments

8.1 The test environment should be relevant to the intended application. Unless otherwise agreed, the environments in 8.3 and 8.4 shall be used.

8.2 Analytical grade chemicals shall be used for the preparation of the corrosive solutions.

8.3 The solution shall be prepared using distilled or deionised water. The minimum resistivity should be 10^5 Ω·cm.

8.4 For alternate immersion, the following test environments are used:

— 0,01 M aqueous sodium chloride solution; the pH of this solution, when freshly prepared for the test, shall be between 5,6 and 6,0;

— 0,01 M Na_2SO_4 solution in pH 4, pH 7,5 to 8,5, pH ≥ 11,5;

— distilled water.

8.5 For continuous immersion, the following test environments are used:

— 0,01 M aqueous sodium chloride solution; the pH of this solution shall be adjusted to ≥ 11,5. The pH shall be checked regularly during the test and adjusted as required;

— 0,01 M aqueous sodium chloride solution at pH 4 and pH 7,5 to 8,5;

— 0,01 M Na_2SO_4 solution, pH 4, pH 7,5 to 8,5, pH ≥ 11,5;

— solution according to ASTM D 1384-96 (148 mg/l Na_2SO_4, 165 mg/l NaCl, 138 mg/l $NaHCO_3$), distilled water.

8.6 Only diluted analytical grade hydrochloric acid/sulphuric acid or sodium hydroxide solutions shall be used to adjust the pH of test solutions. HCl or H_2SO_4 shall be used for chloride and sulfate solutions, respectively.

8.7 The temperature and humidity of the laboratory shall be controlled to ensure that the specimens dry out approximately between consecutive immersion periods during alternate immersion testing.

NOTE Salts on a surface, including $MgCl_2$, can be continuously hydrated during exposure to air despite relatively low humidities.

8.8 Before the freshly prepared solution is used, the temperature of the solution 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 thermal equilibrium.

8.9 The minimum ratio between the volume of test solution and the exposed area of specimens shall be 35 ml/cm^2 for alloys exposed under alternate immersion conditions and under continuous immersion conditions in sodium chloride or sodium sulfate containing solution.

8.10 Evaporation losses shall be minimized by, e.g. using a closed experimental set up, foils, daily additions of water of the required purity (see 8.2).

8.11 The test solution shall be replaced with fresh solution on a weekly basis. At the same time, the portions of the apparatus in contact with the solution shall be cleaned by flushing with water.

9 Loading

9.1 For testing at constant load, the applied load shall be such that the deformation is elastic. The associated stresses shall to be calculated from the formulae given in ISO 7539-4. For specimens containing weldments, they should be tested with and without applied loading in order to assess the effect of residual stresses.

9.2 Constant-strain testing may be conducted as to establish predominantly elastic or plastic deformation (e.g. U-bend test), depending on the testing purposes.

9.3 For constant extension rate tests, testing may be

a) completed to total specimen failure, with ensuing mode of failure assessment to determine stress corrosion cracking susceptibility, or

b) stopped at an intermediate stage to determine the extent of crack initiation or growth.

10 Procedure

10.1 The load should be applied after exposing the specimens to the test environment if possible. Otherwise, the specimens shall be exposed to the test environment as soon as possible after stressing.

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

The air flow past the specimen shall be such as to assist the drying of the specimens between immersion steps.

Drying by forced air blast on the specimens should not be used 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 period.

10.3 In tests under constant load the test duration will depend 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 d and 90 d are employed. For comparability studies the test should be stopped at the same time.

Slow strain rate specimens are typically taken to failure. However, stopping a test at some intermediate stage prior to failure in order to assess the number and depth of cracks is also possible.

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 For slow strain rate tests, experience has shown that for initially plain specimens tested in tension a strain rate of 10^{-6} s^{-1} will be appropriate for the initial test. The absence of stress corrosion cracking from such a test is not necessarily indicative of immunity from stress corrosion cracking in the system studied, since susceptibility is known to be a function of, amongst other parameters, strain rate. Subsequent tests at other strain rates, such as 10^{-5} and 10^{-7} s^{-1}, should be conducted if the initial test produces no evidence of stress corrosion cracking. If specimens are pre-loaded to reduce the total test time during tests at very slow strain rates the comments made in ISO 7539-7:2005, 7.4.3, shall be considered.

10.5 Specimens of dissimilar alloy composition shall not be tested simultaneously in the same solution.

10.6 After exposure, unfailed specimens shall be unloaded, rinsed with distilled water and then cleaned according to 10.7 to remove corrosion products. The same holds for halves of specimens which have failed during testing. Specific care has to be taken to ensure a mild cleaning of the fracture surfaces in order to enable the subsequent evaluation of the failure mode by light or scanning electron microscopy investigation.

10.7 Upon test termination the specimens are first rinsed with distilled water in order to roughly remove the corrosion products and to stop corrosion attack. Corrosion products should then be removed using an appropriate procedure (see ISO 8407). After removal of the corrosion products the specimens are again rinsed with distilled water and then dried with air.

11 Assessment of results

11.1 Test specimens exposed under constant strain, which show significant corrosion after cleaning, can require metallographic examination to assess the presence of cracks. For specimens under constant load, a metallographic examination may be used for failures occurring after more than 10 d, to check if failure is due to stress corrosion or to another form of corrosion. A specimen that reveals only pitting corrosion or pitting plus ductile failure shall not be considered as an SCC failure.

11.2 Optical metallographic examination of a cross-section may be used to distinguish between stress corrosion and other types of corrosion damage.

11.3 For specimens under constant load, scanning electron microscope (SEM) examination of the fracture surface allows failure due to stress corrosion to be distinguished from ductile failure due to mechanical overload. Both types of failures are present when SCC is the origin of failures for specimens under constant load.

11.4 For specimens that were taken to total failure in slow strain rate tests, evidence of stress corrosion cracking is usually apparent from visual examination by low power microscopy for secondary cracking or by change in the failure mode as shown by fractographic assessment of the fracture surface.

12 Expression of results

For specimens tested under slow strain rate conditions, a comparison between identical specimens exposed to the test environment and to an inert environment may be used for assessing the susceptibility to stress corrosion cracking. Increasing susceptibility to cracking is indicated by increasing departure from unity of the ratio

$$\frac{\text{results from specimen in test environment}}{\text{results from specimen in inert environment}}$$

applied to one or more of the following parameters of the same initial strain rate:

a) time to failure;

b) plastic strain to failure;

c) ductility, assessed by, e.g. reduction in area;

d) maximum load achieved;

e) area bounded by nominal stress/elongation curve;

f) percentage of stress corrosion cracking on the fracture surface.

13 Test report

The test report shall include the following information:

a) a reference to this document, including its year of publication, i.e. ISO 20728:2018;

b) the chemical composition or designation of the alloy;

c) product, heat treatment and section thickness of material tested;

d) size and location of specimens;

e) type, size and grain orientation of test specimen and number of replicates;

f) level(s) of stress and method of loading;

g) surface condition of the specimens;

h) test method and test environment (composition and pH, noting any change in pH post-test);

i) test temperature and its variation during testing;

j) duration of test or time to failure of individual specimens, for specimens tested under constant slow strain rate the applied strain rate;

k) results of any metallographic examinations;

l) any unusual features observed and any circumstances or conditions thought likely to affect the results or their validity;

m) any deviation from the method specified;

n) date of the test;

o) the name of the operator and testing organization.