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Corrosion of metals and alloys — Determination of resistance to intergranular corrosion of solution heat-treatable aluminium alloys

Corrosion des métaux et alliages — Détermination de la résistance à la corrosion intergranulaire des alliages d'aluminium aptes au traitement thermique de mise en solution

Reference number
ISO 11846:1995(E)

Foreword

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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.

International Standard ISO 11846 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

Annex A of this International Standard is for information only.

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Corrosion of metals and alloys — Determination of resistance to intergranular corrosion of solution heat-treatable aluminium alloys

1 Scope

1.1 This International Standard specifies the methods of integranular corrosion testing for solution heat-treatable aluminium alloys without protective coatings.

The sensitivity of solution heat-treatable aluminium alloys to intergranular corrosion is a function of the alloy chemical composition, method of manufacturing, solution heat treatment, quench treatment and artificial precipitation hardening (ageing) treatment.

In the naturally aged condition, the sensitivity of solution heat-treatable aluminium alloys to intergranular corrosion is a function primarily of the rate of cooling during quenching over a critical temperature range.

1.2 This International Standard is applicable to cast and wrought heat-treatable aluminium alloys in the form of castings, forgings, plates, sheets, extrusions and semi-finished or finished parts, in order to carry out comparative assessment of alloys of different grades and thickness depending on their chemical composition and other factors, and also to check the thermal processing quality of the tested materials.

The test results provide information to determine the intergranular corrosion resistance and thermal processing quality of the tested materials.

1.3 The test results cannot be regarded as absolute, because they are not applicable to all environments that can be met in service. They are best used in a relative manner, to compare the intergranular corrosion resistance of various heats of solution heat-treatable aluminium alloys.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 8044:1989, *Corrosion of metals and alloys — Vocabulary.*

ISO 8407:1991, *Corrosion of metals and alloys — Removal of corrosion products from corrosion test specimens.*

3 Definitions

For the purposes of this International Standard, the definitions given in ISO 8044 apply.

4 Test specimens

4.1 Sampling

Sampling should be carried out in such a manner as to provide specimens from the most typical areas of the material or the part being tested.

When controlling solution heat treatment, samples are taken from that part of the semi-finished product where cooling rates during quenching are the lowest.

In the case of small parts which are quenched in baskets, the samples are taken from the central part of the basket. If quenching is performed on racks, samples are taken from the upper and lower parts of the rack. If semifinished products, such as tubes, sheets, plates or panels, are vertically quenched the samples are taken from the lower and upper ends. If there are no differences in the cooling conditions, the samples are taken at random.

Samples shall be taken from each solution heat treatment charge.

The location of sampling should be agreed upon between the user and supplier.

4.2 Dimensions, shapes, number of specimens and surface requirements

Specimens can have arbitrary configuration and dimensions, but they should be similar for repetitive tests. The specimen surface area should be between 4 cm^2 and 20 cm^2.

Flat specimens should be cut in such a way that the longer dimension is parallel to the metal working direction.

The specimen surface should maintain the initial condition of the material or finished part, or it should be machined in such a way that the surface roughness R_a is $\leqslant$ 2,5 µm.

Specimens with surface defects (metallurgical or mechanical) should not be tested.

Specimens from clad materials are tested without cladding. The cladding layer is removed from both sides by machining or chemical etching. Etching is carried out in the solutions for dimensional chemical etching or according to 5.2.1. To be certain that all cladding has been removed, it is necessary to remove 0,1 mm more than the thickness of the cladding.

NOTE 1 Specimen heating to above 60 °C during machining is not permitted.

Tests should be carried out on not less than three specimens having the same configuration, dimensions and surface preparation.

5 Surface preparation

5.1 Before testing, specimens are degreased with organic solvent (such as petrol or acetone).

5.2 The specimens are then treated in accordance with one of the methods described in 5.2.1 or 5.2.2.

5.2.1 Specimens are immersed for 2 min to 5 min in sodium hydroxide solution [5 % (*m/m*) to 10 % (*m/m*)] at a temperature of 50 °C to 60 °C, washed in running water, immersed for 2 min in concentrated nitric acid (ρ = 1,4 g/ml) for desmutting, rinsed in running water and then in distilled water, and dried.

5.2.2 Specimens are immersed for 1 min in a solution containing 50 ml of nitric acid (ρ = 1,4 g/ml) and 5 ml of hydrofluoric acid (ρ = 1,15 g/ml) per litre, at a solution temperature of 95 °C ± 2 °C. They are then rinsed in running water, immersed for 2 minutes in concentrated nitric acid (ρ = 1,4 g/ml) for desmutting at room temperature, rinsed in running water and then in distilled water, and dried.

6 Tests

6.1 Naturally aged alloys are tested not earlier than 24 h after quenching. Artificially aged alloys may be tested by this method at any time.

6.2 Tests are carried out according to one of the methods described in 6.2.1 to 6.2.3.

6.2.1 Method A, used to determine quenching quality.

Specimens are immersed for 6 h in a solution containing 57 g/l ± 1 g/l of sodium chloride and 10 ml ± 1 ml of hydrogen peroxide [30 % (*V/V*)] at a temperature of 30 °C ± 3 °C. After testing, the specimens are rinsed in running water and are allowed to dry. Corrosion products may be removed with a non-metallic brush during rinsing and/or preferrably dipping in concentrated nitric acid [70 % (*m/m*) HNO_3, ρ = 1,4 g/ml] for only a few minutes just sufficient to dissolve corrosion products, followed by rinsing thoroughly with tap water. (See ISO 8407.)

6.2.2 Method B, used to compare the intergranular corrosion resistance of various solution heat-treatable aluminium alloys, depending on chemical composition and heat treatment.

Specimens are immersed for 24 h in a solution containing 30 g/l sodium chloride and 10 ml ± 1 ml of concentrated hydrochloric acid (ρ = 1,19 g/ml) at room temperature. After testing, the specimens are rinsed in running water and then in distilled water and are allowed to dry. Corrosion products may be re-

moved with a non-metallic brush during rinsing and/or preferrably dipping in concentrated nitric acid [70 % (*m/m*) HNO_3, $\rho = 1,4$ g/ml] for only a few minutes just sufficient to dissolve corrosion products, followed by rinsing thoroughly with tap water. (See ISO 8407.)

6.2.3 Method C, used to estimate the sensitivity of aluminium-lithium alloys to intergranular corrosion.

The principle involves anodic polarization of specimens in sodium chloride solution up to the potential at which the alloy shows intergranular corrosion susceptibility and to the exposure at this potential (E_{icc}). (See figure 1.)

NOTE 2 Method C can also be used for other alloy systems.

Tests are carried out in a thermostatically controlled electrochemical cell (which includes test, auxiliary, and reference electrodes). The test solution is 0,01 % (*m/m*) sodium chloride. A potentiostat is used to polarize the test electrode at controlled scan rates and at the E_{icc} potential.

The test electrode is a specimen with a mechanically or electrochemically polished surface area of 1 cm^2.

The auxiliary electrode is a platinum electrode, and the reference electrode is either calomel or silver/silver chloride.

First, the anodic polarization curve is plotted for one specimen by scanning the potential from a cathodic value of $E = -1,16$ V at a scan rate of 0,6 V/h to the pitting potential, E_{pf}. (The pitting potential is a potential at which the density of current is increased by at least one order in the process of anodic polarization.) Another specimen is then immersed in the same cell, allowed to rest for 5 min, and the potential moved to

$$E_{icc} = E_{pf} + 20 \text{ mV}$$

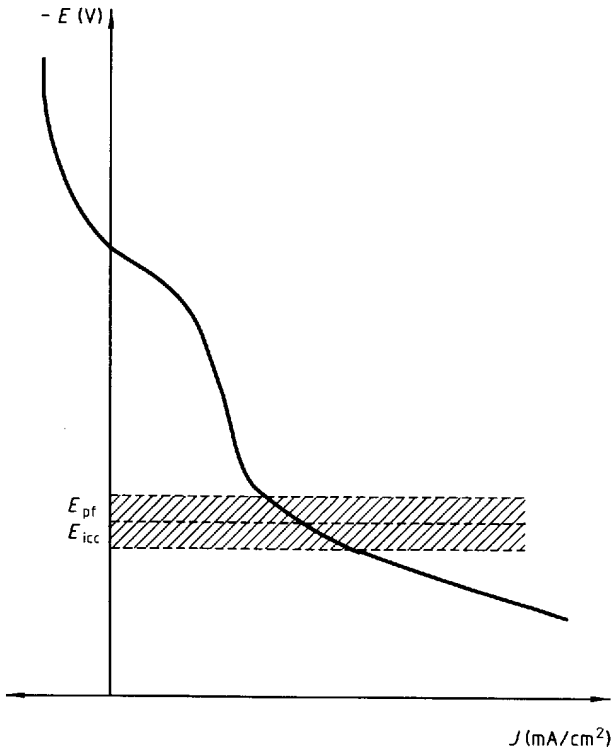
Exposure at this potential is continued as follows:

— for copper-containing alloys: 15 min ± 1 min;

— for copper-free alloys: 90 min ± 5 min.

After the tests the specimens are taken out of the cell, washed in distilled water, dried and metallographically examined.

6.3 Solutions are prepared using distilled or deionized water with a conductivity not greater than 10 µS/cm (see ISO 3696) just before testing. To prepare the solutions, analytical grades of chemicals are used.



E_{pf} = pitting formation potential

$E_{icc} = E_{pf} + 20$ mV

Intercrystalline corrosion region

Figure 1 — Typical anodic polarization curve

6.4 The ratio of the solution volume to the total specimen area should be not less than 5 cm^3/cm^2.

6.5 The specimens are placed in the solution in such a way that they do not touch each other and the vessel walls. The solution level above the specimens should be not less than 20 mm above the upper edge, and it should be the same for all the specimens. It is not permissible to test specimens of different alloy systems in the same solution.

6.6 Tests are carried out in glass vessels or in vessels made from inert organic materials.

7 Metallographic examination

Each tested specimen is examined at × 5 magnification, and the two zones with the most serious corrosion attack are marked. The sections are cut from these zones and are prepared for microscopic

examination. The sections are taken normal to the largest surface of the tested specimen and should be taken far enough from the edges of the test specimen to avoid areas of edge corrosion attack.

Cross-sections are examined by microscope in the unetched condition with magnifications of × 100 to × 500. If there is any uncertainty that the corrosion is truly intergranular, a light metallographic etch may be used to identify actual grain boundaries.

8 Interpretation of the results

Interpretation of the sensitivity of solution heat-treatable aluminium alloys to intergranular corrosion is based on the type (etching, pitting, or intergranular corrosion), depth and relative extent of the attack (the length along the surface of the metallographic cross-section) and is expressed as a percentage.

When used to judge the quality of solution heat-treatment, the acceptable susceptibility to intergranular corrosion is agreed upon between the supplier and the user.

9 Test report

The test report should include the following information:

a) designation and chemical composition of the alloy;

b) type of semi-finished product or part;

c) method of manufacturing of product or part;

d) heat treatment;

e) surface condition;

f) specimen sizes;

g) applied test method (referring to this International Standard);

h) period of exposure;

i) rating criteria of the alloy resistance to intergranular corrosion, rating number.

Annex A
(informative)

Bibliography

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