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STANDARD

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**Corrosion of metals and alloys — Anodic
test for evaluation of intergranular
corrosion susceptibility of heat-treatable
aluminium alloys**

*Corrosion des métaux et alliages — Essai anodique pour l'évaluation de
la sensibilité à la corrosion intergranulaire des alliages d'aluminium
aptés au traitement thermique*

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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 15329 was prepared by Technical Committee ISO/TC 156, *Corrosion of metals and alloys*.

Corrosion of metals and alloys — Anodic test for evaluation of intergranular corrosion susceptibility of heat-treatable aluminium alloys

1 Scope

This International Standard specifies an electrochemical method to determine susceptibility to intergranular corrosion of solution-heat-treatable aluminium alloys, that is 2XXX, 6XXX, 7XXX and 8XXX alloys, without protective coatings and in various ageing conditions.

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 a 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 help to determine the intergranular corrosion resistance and thermal processing quality of the tested materials (see Clauses 8 and 9).

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

ISO 8044:1999, *Corrosion of metals and alloys — Basic terms and definitions*

ISO 11846:1995, *Corrosion of metals and alloys — Determination of resistance to intergranular corrosion of solution heat-treatable aluminium alloys*

3 Terms and definitions

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

4 Principle

The method is based on the principle that, if an aluminium alloy is susceptible to intergranular corrosion, this susceptibility will show as a breakdown during the anodic polarization of the alloy when exposed to solutions containing chloride ions.

The sensitivity of solution-heat-treatable aluminium alloys to intergranular corrosion depends on the alloy 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.

Regardless of the type of pitting initiation and development zones (along the grain boundaries or through the body of a crystal), depassivation (disturbance of passive state of the metal) occurs at the attaining of pitting initiation potential. The development of intergranular corrosion occurs more frequently at the potential which is somewhat more positive compared to the pitting-initiation potential.

Historically, acceleration of intergranular corrosion tests have tended toward arbitrary, and possibly extreme conditions, including impressed electrical currents (galvanostatic) or impressed electrode potentials (potentiostatic). Such testing can be improved by selecting the electrochemical force, with consideration of the relative anodic characteristics of the material in relationship with

- a) the relative cathodic phases of the material, and
- b) the chemical composition of the test medium.

This test method starts with the anodic polarization of a specimen to determine the subsequent impressed potential. Like any other accelerated test, the test results must be correlated with the service performance of the materials being tested.

Metallographical examination is needed to determine the localised corrosion mode.

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

5 Specimens

5.1 Test specimens

Sampling should be carried out in order to provide representative specimens from the most typical areas of the material or the part being tested.

When controlling solution heat treatment, test specimens 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 semi-finished 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 (see ISO 11846).

Samples shall be taken from each solution-heat-treatment charge.

The location of sampling and three-dimensional grain structure should be agreed upon between the user and supplier.

5.2 Dimensions, shapes, number of specimens and surface requirements

Specimens can have arbitrary configuration and dimensions, but they should be similar for repetitive tests.

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

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

6 Surface preparation

6.1 Before testing, specimens should be mechanically ground and degreased with an organic solvent (hydrocarbon, with a boiling point between 60 °C and 120 °C) using a clean soft brush or an ultrasonic cleaning device, carrying out the cleaning in a vessel full of solvent. After cleaning, the specimens should be rinsed with a fresh solvent, dried and stored in a desiccator for 1 h.

6.2 Pre-treatment may be carried out according to ISO 11846.

7 Procedure

7.1 Naturally aged alloys are tested 24 h after quenching. Artificially aged alloys may be tested by this method at any time.

7.2 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.

7.3 The ratio of the solution volume to the total specimen area should be not less than 50 ml/cm², and a fresh solution should be used for each test.

7.4 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 should be not less than 20 mm above the upper edge of the specimens and should be the same for all specimens. It is not permissible to test specimens of different alloy systems in the same solution.

7.5 Tests are carried out in glass vessels, or in vessels made from inert materials.

7.6 Tests are carried out according to the following procedure:

Tests are carried out in a thermostatically controlled, electrochemical cell (which includes test, auxiliary, and reference electrodes), at between 18 °C and 25 °C. The test solution is sodium chloride with a mass fraction of 0,1 %. A potentiostat is used to polarize the test electrode at controlled scan rates and at the E_{ic} potential.

The test electrode is a specimen with a mechanically polished surface area not less than 1 cm². The surface roughness $R_a \leq 1 \mu\text{m}$.

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 $E = -2,00 \text{ V}$ to the hydrogen electrode at a scan rate of 50 mV/min to the pitting initiation potential, E_{pi} . The potential for pitting initiation is the potential after which the density of current is increased by at least one order of magnitude in the process of anodic polarization (see ISO 8044). Another specimen is then immersed in the same cell, allowed to rest for 5 min, and the potential is moved to:

$$E_{ic} = E_{pi} + 50 \text{ mV}$$

Exposure at this potential is continued as follows:

- for copper-containing alloys (range 0,25 % to 6,5 % Cu): 10 min \pm 1 min;
- for copper-free alloys (maximum 0,25 % Cu): 40 min \pm 5 min.

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

8 Metallographic examination

Each tested specimen is examined at $\times 5$ to $\times 20$ magnification, and one or two zones with the most serious corrosion attack are marked. The sections for metallographic analysis are cut through these zones and are prepared for microscopic examination.

9 Assessment of results

Interpretation of the sensitivity of solution-heat-treatable aluminium alloys to intergranular corrosion is based on the type (pitting or intergranular), depth, and relative extent of the attack.

At first, the type of corrosion should be determined according to the references (see Figure 2, A, B, C, D). References A and B represent pitting corrosion, i.e. the cases when intergranular corrosion susceptibility is absent. References C and D represent intergranular corrosion. In this case, its rating should be classified according to Table 1 on the basis of maximal corrosion-damage depth.

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

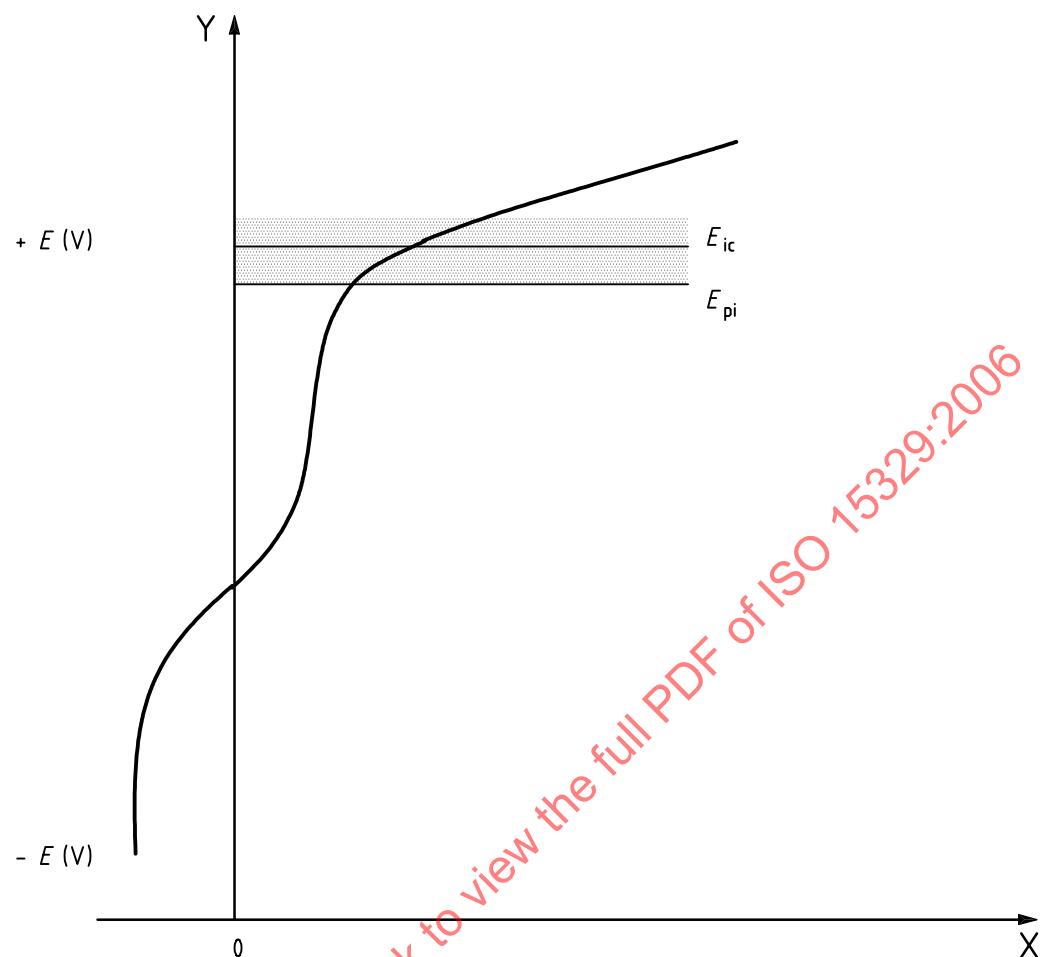
10 Test report

The test report shall include the following information:

- a) a reference to this International Standard: ISO 15329;
- b) designation and composition of the alloy;
- c) type of semi-finished product or part;
- d) method of manufacturing of product or part;
- e) heat treatment;
- f) surface condition;
- g) specimen sizes;
- h) pitting potential;
- i) period of exposure;
- j) current at the pitting potential at the end of testing;
- k) type of corrosion;
- l) rating criteria of the alloy resistance to intergranular corrosion and rating number.

Table 1 — Intergranular corrosion ratings for aluminium alloys (see [2] in the Bibliography)

Corrosion resistance	Rating	Maximum corrosion depth μm
Excellent	1	0
Good	2	100
Satisfactory	3	100 to 200
Poor	4	200 to 400
Very poor	5	More than 400

**Key**

X current density
Y potential

Figure 1 — Typical anodic polarization diagram