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Corrosion of metals and alloys — Aqueous corrosion testing of zirconium alloys for use in nuclear power reactors

Corrosion des métaux et alliages — Essais de corrosion aqueuse des alliages de zirconium utilisés dans les réacteurs nucléaires

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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.

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 are 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 is not 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*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 262, *Metallic and other inorganic coatings, including for corrosion protection and corrosion testing of metals and alloys*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 10270:1995), which has been technically revised. It also incorporates the Technical Corrigendum ISO 10270:1995/Cor 1:1997. The main changes compared with the previous edition are as follows:

— the references have been updated;

— in Clause 4, two sentences have been added: "The tests in water is performed at 18,6 MPa. The pressure is also determined by the contractor's requirements.";

— in 5.2, "the test" has been replaced by "the measurement to avoid erroneous results from differing corrosion behaviour of the materials";

— in 7.1, "welding grade" has been replaced by "of purity 999,9 ml/l or higher",

— in 11.1, d), "1 day" has been replaced by "24 h"; and "at 18,6 MPa" has been added after "at 360 °C in water".

— in 12.1, a sentence has been added: "Post-heat treatment, the coupons are inspected for any residual oxide and prepared (e.g. etched) in line with standard coupons";

— in 12.3.4, a sentence has been added: "However, for the product acceptance test, it is better to keep the control coupons as evidence of test effectiveness.";

— in 13.3.1, "±3 °C for steam tests, and ±6 °C for water tests" has been replaced by "±3 °C for steam tests and water tests";

— in 13.3.3, "3 days or 14 days" has been replaced by "72 h or 336 h";

— in 14.2, a sentence has been added: “Mass gain measurements are taken in triplicate for each specimen and a mean value calculated”;

— in 14.3.1, a sentence has been added: “The separators are such as not to induce Galvanic interaction between the samples and the separators”; “zirconium or zirconium alloy” has been added before “stainless steel”;

— in Clause 16, “including its number and year of publication” has been added before “this document”;

— in A.3.1, “3 % (m/m) of hydrofluoric acid (7.7), 39 % (m/m) of nitric acid (7.8)” has been replaced by “40 g/kg ± 20 g/kg of hydrofluoric acid (7.7), 450 g/kg ± 50 g/kg of nitric acid (7.8)”, and a sentence has been added: “A different acid ratio may be chosen to satisfy the requirement of a smooth and shining surface given in 13.4.1;

— in A.3.2, “9 % (m/m) ± 1 % (m/m)” has been replaced by “90 g/kg ± 10 g/kg” and “30 % (m/m) ± 5 % (m/m)” has been replaced by “300 g/kg ± 50 g/kg”;

— in A.4.2,“25 % (V/V)” has been replaced by “250 ml/l” and “50 % (V/V)” has been replaced by “500 ml/l”;

— in Clause A.6, the following text has been added: “or placed into the oven at a recommended temperature of 60 °C to 70 °C for 1 h" and “The specimens need to be cooled to room temperature after removing from the oven”.

Any feedback or questions on this document are 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 metals and alloys — Aqueous corrosion testing of zirconium alloys for use in nuclear power reactors

WARNING — This document can involve the use of hazardous materials, operations and equipment (see Clause 9). It is the responsibility of the user of this document to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This document specifies:

a) the determination of mass gain;

b) the surface inspection of products of zirconium and its alloys when corrosion is tested in water at 360 °C or in steam at or above 400 °C;

c) the performance of tests in steam at 10,3 MPa.

This document is applicable to wrought products, castings, powder metallurgy products and weld metals.

This method has been widely used in the development of new alloys, heat-treating practices and for the evaluation of welding techniques. It is applicable for use in its entirety to the extent specified for a product acceptance test, rather than merely a means of assessing performance in service.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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 https://www.electropedia.org/

3.1
etching
process for the removal of surface metal by action of acids in water

3.2
control coupon
zirconium alloy specimen of known performance used to monitor the validity of the test

3.3
high mass gain coupon
zirconium alloy specimen that has been specially heat-treated to produce a mass gain higher than the maximum given in materials acceptance specifications and which is used for verifying the severity of the test procedure

4 Principle

Specimens of zirconium or its alloys are exposed to high-pressure water or steam at elevated temperatures for 72 h or 336 h. The corrosion is normally measured by the gain in mass of the specimens and by the appearance of an oxide film on the specimen surfaces. In some instances, such as weld evaluation, mass gain measurements are either impractical to make or are not required. When so specified, the appearance of the specimen is the sole criterion for acceptance. The test pressure in steam is 10,3 MPa. The tests in water is performed at 18,6 MPa. The pressure is also determined by the contractor's requirements.

5 Significance

5.1 Specimens are normally tested after careful etching and rinsing. Specimens with as-manufactured surfaces may also be tested without further surface removal.

5.2 When tubing with a second material clad on the surface is to be tested, the internal cladding is removed prior to the measurement to avoid erroneous results from differing corrosion behaviour of the materials.

6 Interference

Autoclave loads that have one or more specimens showing gross oxidation can, by contamination of the environment, affect results of other specimens in the autoclave.

7 Reagents and materials

During the test, unless otherwise stated, only reagents of recognized analytical grade and only water as described in 7.4 or 7.5 are used.

7.1 Argon gas, of purity 999,9 ml/l or higher.

7.2 Nitrogen gas, for purging or controlling oxygen content.

7.3 Argon-hydrogen mixture, for purging to remove dissolved oxygen.

7.4 Grade A water, purified water having an electrical resistivity of not less than 1 MΩ·cm at 25 °C as measured before the start of the test.

7.5 Grade B water, deionized or demineralized water having an electrical resistivity of not less than 0,5 MΩ·cm at 25 °C. Grade A water can be used instead of grade B water.

7.6 Detergents and solvents, for specimen cleaning, including ethanol and acetone.

7.7 Concentrated hydrofluoric acid (HF).

7.8 Concentrated nitric acid (HNO_3).

7.9 Concentrated sulfuric acid (H_2SO_4).

7.10 Control coupon.

7.11 High mass gain coupon.

8 Apparatus

The apparatus consists of equipment for:

a) etching the specimen when required;

b) measuring the specimens' surface area and mass, the water resistivity and pH, the test temperature and pressure, and the etch and rinse temperature;

c) performing the water or steam corrosion tests at elevated temperatures and pressures.

8.1 Etching equipment, comprising an acid bath, a running water rinse and a deionized water rinse needed for proper metal removal and stain-free rinsing. Polyethylene or polypropylene tanks are commonly used with a bottom feed for running water rinses. Specimen hangers are generally made of type 300 series stainless steel. When many specimens are processed, a mechanical dipper for the etching process is useful.

8.2 Autoclaves, constructed of type 300 series stainless steel or nickel base alloys such as UNS grade N06600 or N066901. The autoclave is fitted with devices for measurement and control of pressure and temperature, safety devices and venting valves. Control systems for pressure and temperature are adequate to meet the requirements of this document. Sample holders and other internal accessories are also constructed of type 300 or type 400 series stainless steel, or nickel-base alloys such as UNS[1)] grade N06600 or N06690.

8.3 Specimen size measuring equipment, accurate to 50 µm.

8.4 Analytical balance, accurate to 0,1 mg.

9 Hazards

9.1 The chemicals used in preparing specimens for this test are hazardous. Detailed information on the safe handling of organic compounds, acids and products of zirconium and its alloys are obtained from competent sources.

9.2 High-temperature, high-pressure autoclave operation is in accordance with the manufacturer's instructions.

9.3 Hydrogen gas used for the addition to the autoclave steam supply is handled in accordance with the guidelines for explosives and inflammable substances.

9.4 Cold water is not added directly to the autoclave vessel in order to accelerate cooling upon the completion of testing.

10 Sampling, test specimens and test units

10.1 The size and the quantity of the test specimens, the method of selection, surface preparation and test acceptance criteria are specified in the product specification or by agreement between the purchaser and the seller as stated in the purchase contract. The total surface area for each specimen is a minimum of 10 cm^2.

10.2 Each specimen and control coupon is individually identified.

1) UNS grades N06600 and N06690 are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

11 Preparation of apparatus

11.1 General requirements for new or reworked autoclaves (8.2) and parts of autoclaves previously used for testing materials other than in accordance with this document are as follows.

a) Before specimens are tested in a new or reworked autoclave, or in one having new valves, tubing, gaskets, etc. which are in contact with the test specimen, clean the apparatus thoroughly, wipe with ethanol or acetone (7.6) and rinse twice with grade B water (7.5).

b) Dry the autoclave or auxiliary equipment by vacuum cleaning, or drain and wipe with a clean, lint-free cloth and inspect carefully to ensure freedom from contamination. There is no visible contamination, such as lubricant, residues, dust or dirt, loose oxides or rust, or an oil or grease film on the water surface, internal surface, gasket or head surfaces.

c) Clean all new and reworked fixtures and jigs to be used in the autoclave and rinse in hot grade B water.

d) Autoclave the fixtures and jigs for at least 24 h at 400 °C in steam at 10,3 MPa or, preferably, at 360 °C in water at 18,6 MPa.

e) Inspect the parts for corrosion products. If corrosion products are found or electrical resistivity of the residual water after the test measures less than 0,1 MΩ·cm, clean and autoclave the parts again.

11.2 General requirements for autoclaves and parts in continuous use for corrosion testing in accordance with this document are as follows.

a) All autoclaves, fixtures, parts and jigs that have been in continuous use and have shown satisfactory behaviour in prior tests are rinsed with grade B water.

b) The fixtures and jigs for corrosion products are inspected after each test and items showing loose corrosion products are re-worked and re-prepared.

12 Calibration and standardization

12.1 High mass gain coupon preparation

High mass gain coupons (7.11) is selected from a previously tested lot. For Zircaloy type alloys, the selected material is specially heat-treated prior to exposure in order to produce the desired mass gain in the autoclave test. Heating for 8 h at 900 °C and cooling to 300 °C at a rate not exceeding 3,3 °C/min in an inert atmosphere [e.g. Ar (7.1)] or vacuum is a suggested procedure, but alternative procedures can be used. This treatment is used in the case of Zircaloy type alloys. Other alloy types can require different heat-treatment. Post-heat treatment, the coupons are inspected for any residual oxide and prepared (e.g. etched) in line with standard coupons.

12.2 Autoclaves

12.2.1 Prior to use for product acceptance testing, an autoclave is profiled thermally as given in 12.4.2 and demonstrates acceptability by testing at least three control coupons: one each at the top, middle and bottom of a useful volume. The test results are incorporated in the certification document for the autoclave acceptance test. When desired, high mass gain coupons may also be used.

The new or used autoclave is considered acceptable if each control coupon mass gain is reproducible within the previously established control coupon mean mass gain, ±3 standard deviations.

12.2.2 The control coupon lot (7.10) and, when desired, the high mass gain coupon lot (7.11) mass gain mean and standard deviation are established by a minimum of one autoclave test as follows.

a) Randomly select 12 specimens from either the control coupon lot or the high mass gain coupon lot or both.

b) Prepare all specimens in accordance with 13.4 in order to meet the pre-test requirements of this document.

c) Locate the 12 or 24 specimens in a fixture or jig (see Figure 1) and place the fixture or jig inside the useful volume of the autoclave.

d) Complete the steam or water corrosion test in accordance with any one of the four methods in 14.3.

e) Remove the specimens and weigh in accordance with the requirement of this document.

f) Calculate and establish the mass gain mean and standard deviation [(n − 1) method] of each set of coupons for the test method used.

For the product acceptance tests, the mean value and standard deviation for the control coupons is the value established as described above or it is calculated periodically using all accepted values determined over the preceding three-month period but not less than 21 values.