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ISO/DIS 10270

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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

ICS: 77.060; 27.120.10

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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 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 on 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 the following URL: www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 10270:1995), which has been technically revised.

The main changes compared to the previous edition are as follows:

— Chapter 2 The first paragraph is replaced by "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." And"1990" is replaced by "2012" for the latest vision of ISO 5814.

— Chapter 3 The title of " Definitions" is replaced by "Terms and definitions". And the first paragraph is replaced by:

"For the purposes of this document, the following terms and definitions apply.

The list below is always included after each option:

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

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

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

— Chapter 4 Two sentence are added that "the tests in water shall be performed at 18.6 MPa (2 697 psi)" and"The pressure should also be determined by the contractor requirements" in the end.

— Chapter 5.2 The word of " internal" is added before "cladding" .

— Chapter 7.1 The words of " welding grade" is replaced by "purity is 99.99 % or higher".

— Chapter 7.3 The word of "dissolved" is added before "oxygen".

— Chapter 8.1 The bracket is added for the superscript number “1”.

— Chapter 11.1.2 The unit of “1 day” is replaced by “24 h”; and the words of “at 18.6 MPa” is added after “at 360 °C in water”.

— Chapter 12.1 The word of “Zircalloy” is replaced by “Zircaloy”.

— Chapter 12.3.4 It is added that “However, for product acceptance test, it is better to keep control coupons as evidence of test effectiveness.” in the end.

— Chapter 14.3.2.1 The tile of “The Relationship between Temperature and the Specific Volumes of Water” is added for Table 1.

— Annex A.3.1 “3 % (m/m) of hydrofluoric acid (7.7), 39 % (m/m) of nitric acid (7.8)” is instead by “4 % (m/m) ± 2 % (m/m) of hydrofluoric acid (7.7), 45 % (m/m) ± 5 % (m/m) of nitric acid (7.8)”. And the sentence is added that “If the different acid ratio is chose to satisfy the requirement of smooth and shining surface as mentioned in 13.4.1, it is acceptable”in the end.

— Annex A.6 It is added that“ or be placed into the oven, the condition of 60 - 70 °C for one hour is recommended at the oven ”after “dust and acid fumes”. The sentence is added that “The specimens need to be cooled to room temperature after removing from the oven” in the end.

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.

Introduction

The nuclear industry has been developed in recent years, but the latest standard was published in 1997, and not revised until now. There are some problems in the standard after 22 years. The revised standard is more suitable for the application in Nuclear Industry. The revised standard will be more professional and precise, the customers will understand the standard very well and avoid the misunderstanding.

The problems are :

1) Pressure: There is no pressure in water at 360 °C. In general, saturated vapor pressure of water is 18.6 MPa 360 °C. In the world, the pressure in water at 360 °C is only 18.6 MPa for the corrosion of zirconium alloys. Many references also prove that the pressure is 18.6 MPa for water at 360 °C. The pressure shall be added in the text.

2) Re-use: In Chapter 12.3.4, Control coupons may be re-used after mechanical removal of oxide film followed by etching as described in 13.4.1. Actually, the oxide is very dense and hard to be removed. The weight gain is not real if metal oxides were still on the surface of the control coupons. To ensure the experiment effective, it is added that “however, for product acceptance test, it is better to keep control coupons as evidence of test effectiveness” at the end of the paragraph.

3) Unit: The unit is hour in Chapter 4, but the unit is days in Chapter 13.3.3. Both sentences express the same meaning, but the unit is different. The unit of hour is applied for the whole text.

4) The range of acid: 3 % (m/m) of hydrofluoric acid and 39 % (m/m) of nitric acid are mentioned in Appendix 3.1. But 9 % (m/m) ± 1 % (m/m) of hydrofluoric acid, 30 % (m/m) ± 5 % (m/m) nitric are mentioned in Appendix 3.2. So 4 % (m/m) ± 2 % (m/m) of hydrofluoric acid, 45 % (m/m) ± 5 % (m/m) of nitric acid are chosen for the range of acid bases on the experiments in Appendix 3.1.

5) Dry method:The oven is often applied for drying the samples besides lint-free cloth or dry air. The oven is more efficiency for the treatment of lots of samples.

Identification of patent holders, if any.

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

WARNING — — This International Standard may involve the use of hazardous materials, operations and equipment (see clause 9). It is the responsibility of whoever uses this International Standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope *(mandatory)*

This International Standard specifies:

a) the determination of mass gain;

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

c) that the tests in steam shall be performed at 10.3 MPa (1 500 psi);

This International Standard 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, and should be utilized 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 *(mandatory)*

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.

Members of IEC and IS0 maintain registers of currently valid International Standards.

ISO 5813:1983, *Water quality — Determination of dissolved oxygen — lodometric method.*

ISO 5814:2012, *Water quality — Determination of dissolved oxygen — Electrochemical probe method.*

3 Terms and definitions *(mandatory)*

For the purposes of this document, the following terms and definitions apply.

The list below is always included after each option:

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

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

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

3.2
control coupons
Zirconium alloy specimens of known performance used to monitor the validity of the test.

3.3
high mass gain coupons
Zirconium alloy specimens that have been specially heat-treated to produce a mass gain higher than the maximum given in materials acceptance specifications and which are 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 shall be the sole criterion for acceptance. The test pressure in steam shall be 10.3 MPa (1 500 psi); the tests in water shall be performed at 18.6 MPa (2 697 psi). The pressure should also be determined by the contractor 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 shall be removed prior to the test.

6 Interference

Autoclave loads that have one or more specimens showing gross oxidation may, 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 shall be used.

7.1 **Argon gas**, purity is 99.99 % 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 coupons, as defined in 3.2.

7.11 High mass gain coupons, as defined in 3.3.

8 Apparatus

8.1 General

The apparatus shall consist of equipment for:

a) etching the specimens when required;

b) measuring the specimens[1] surface area and mass, the water resistivity and pH, the test temperature and pressure, the etch and rinse temperature;

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

8.2 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.

8.3 Autoclaves, constructed of type 300 series stainless steel or nickel base alloys such as UNS grade NO6600 or NO6690 and manufactured to conform to government regulations governing unfired pressure vessels. 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 shall be adequate to meet the requirements of this International Standard. 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 grade NO6600 or NO6690.

8.4 Specimen size measuring equipment, accurate to 50 µm.

8.5 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 safe handling of organic compounds, acids and products of zirconium and its alloys should be obtained from competent sources.

9.2 High-temperature, high-pressure autoclave operation must be in accordance with government regulations and manufacturer's instructions.

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

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