BRITISH STANDARD

BS EN ISO 10270:2008

Incorporating corrigendum July 1997

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

ICS 27.120.10; 77.060

British Standards

National foreword

This British Standard is the UK implementation of EN ISO 10270:2008. It is identical with ISO 10270:1995, incorporating corrigendum July 1997. It supersedes BS ISO 10270:1995 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee ISE/NFE/8, Corrosion of metals and alloys.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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EUROPEAN STANDARD
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EN ISO 10270

April 2008

ICS 77.060; 27.120.10

English Version

Corrosion of metals and alloys - Aqueous corrosion testing of zirconium alloys for use in nuclear power reactors (ISO 10270:1995/Cor 1:1997)

Corrosion des métaux et alliages - Essais de corrosion aqueuse des alliages de zirconium utilisés dans les réacteurs nucléaires (ISO 10270:1995/Cor 1:1997)

Korrosion von Metallen und Legierungen - Korrosionsprüfung in wässrigen Lösungen für in Kernreaktoren angewendete Zirkoniumlegierungen (ISO 10270:1995/Cor 1:1997)

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Foreword

The text of ISO 10270:1995/Cor 1:1997 has been prepared by Technical Committee ISO/TC 156 “Corrosion of metals and alloys” of the International Organization for Standardization (ISO) and has been taken over as EN ISO 10270:2008 by Technical Committee CEN/TC 262 “Metallic and other inorganic coatings” the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2008, and conflicting national standards shall be withdrawn at the latest by October 2008.

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Endorsement notice

The text of ISO 10270:1995/Cor 1:1997 has been approved by CEN as a EN ISO 10270:2008 without any modification.

Contents

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

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

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

ISO 5814:1990, *Water quality — Determination of dissolved oxygen — Electrochemical probe method.*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

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).

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 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*, welding grade.

7.2 *Nitrogen gas* for purging or controlling oxygen content.

7.3 *Argon-hydrogen mixture* for purging to remove 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', surface area and mass, the water resistivity and pH, the test temperature and pressure, the etch and rinse temperature;

c) performing 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 N06600 or N06690 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 N06600 or N06690.

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.

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 shall be specified in the product specification or by agreement between the purchaser and the seller as stated in the purchase contract. Total surface area for each specimen shall be a minimum 10 cm^2.

10.2 Each specimen and control coupon shall be individually identified.

11 Preparation of apparatus

11.1 General requirements for new or reworked autoclaves (**8.3**) and parts of autoclaves previously used for testing materials other than in accordance with this International Standard are as follows.

11.1.1 Before specimens are tested in a new or re-worked 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**). 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 should be 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.

11.1.2 Clean all new and reworked fixtures and jigs to be used in the autoclave and rinse in hot grade B water. Autoclave the fixtures and jigs for at least 1 day at 400 °C in steam at 10,3 MPa or, preferably, at 360 °C in water. 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 under this International Standard are as follows.

All autoclaves, fixtures, parts and jigs that have been in continuous use and have shown satisfactory behaviour in prior tests shall be rinsed with grade B water. The fixtures and jigs for corrosion products shall be inspected after each test and items showing loose corrosion products, shall be re-worked and re-prepared.

12 Calibration and standardization

12.1 High mass gain coupon preparation

High mass gain coupons (**7.11**) shall be selected from a previously tested lot. The selected material shall be 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 or vacuum is a suggested procedure, but alternative procedures can be used. This treatment shall be used in the case of Zircalloy type alloys. Other alloy types may require different heat treatment.

12.2 Autoclaves

12.2.1 Prior to use for product acceptance testing, an autoclave shall be profiled thermally as in **12.4.2** and shall demonstrate acceptability by testing at least three control coupons, one each at the top, middle and bottom of a useful volume. The test results shall be 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 shall be established by a minimum of one autoclave test as follows.

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

Prepare all specimens in accordance with **13.4** in order to meet the pretest requirements of this International Standard.

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.

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

Remove specimens and weigh in accordance with the requirement of this International Standard.

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

NOTE 1 For the product acceptance tests, the mean value and standard deviation for the control coupons may be the value established as described above or it may be calculated periodically using all accepted values determined over the preceding three-month period but not less than 21 values.

12.2.3 An alternative method for establishing the mass gain mean and standard deviation is as follows.

Expose the control coupons to be used in three different tests. In each test a minimum of one specimen should be exposed in each of the top, middle and bottom positions of the autoclave. Determine the mass gain.

The mean value for control coupons is the mean for the three tests and three positions.

The standard deviation for the control coupon lot is calculated by the $(n-1)$ method using the data from all of the control coupons taken from the same material lot.

12.3 Use of control coupons

12.3.1 Each autoclave run used for acceptance of product shall contain at least three control coupons with one at the top, one at the middle and one at the bottom of the specimen load.

12.3.2 The control coupons may be as manufactured or etched before testing, but if etched, the surfaces shall exhibit no stains, pits or areas of abnormal etching attack.

12.3.3 An autoclave test shall be considered acceptable only if each post-test control coupon mass gain is not less than the established mean value – 3 standard deviations and the visual appearance of each control coupon is equal to or better than the product acceptance standard. If a control coupon post-test mass gain exceeds the mean value + 3 standard deviations, or the specified mass gain value and one or more test specimens from the corresponding location in the autoclave fail to meet the mass gain acceptance criterion, the failed specimen(s) may be discarded and a new test made to determine conformance.

12.3.4 Control coupons may be re-used after mechanical removal of oxide film followed by etching as described in **13.4.1**.

12.4 Calibration

12.4.1 The temperature measurement and recording systems used to determine conformance shall be calibrated at least every six months and shall not deviate more than ± 3 °C from the calibration reference materials traceable to national standards.

12.4.2 Vertical thermal profiles of the autoclaves at the test temperatures shall be made at least once in each six-month period, or whenever the heaters or the control thermocouples are adjusted or replaced. The axial extent of the autoclave used for performing the product acceptance testing shall be restricted to the volume shown to be within ± 3 °C of the recorded autoclave temperature, after temperature compensation for calibration of the thermocouples. This volume is considered the useful volume. The profile thermocouples may be located at the centre or near the radial extremity of the autoclave volume.

12.4.3 Pressure-measuring devices shall be calibrated annually and the recorded reading shall be within ± 0,35 MPa of the calibrated device over the range used for testing.

13 Conditioning

13.1 Test water quality

The water used to conduct the corrosion test shall be grade A water (**7.4**) and have an oxygen content not exceeding 45 pg/g. The oxygen content specification may be met either by direct measurement using the appropriate method specified in ISO 5813 or ISO 5814 or by the use of venting method A or B specified in **14.3**.

13.2 Autoclave load restrictions

The surface area of specimens loaded in a static autoclave should not exceed 0,1 m^2/l of autoclave volume.

13.3 Test conditions

13.3.1 *Temperature*

The recorded temperature within the volume used for testing shall be the specified value ± 3 °C for steam tests, and ± 6 °C for water tests.

13.3.2 *Pressure*

The recorded pressure shall be as specified ± 0,7 MPa for steam tests and ± 1,4 MPa for water tests.

13.3.3 *Time*

The recommended exposure times are 3 days or 14 days. The exposure time tolerance at the specified temperature and pressure shall be $^{+8}_{0}$ h. The time at specified conditions need not be continuous.

13.3.4 *Tests*

Tests where temperature or pressure limits or both are exceeded for not more than 10 % of the nominal test time, but where control coupons indicate satisfactory behaviour may, at the discretion of the test laboratory, be deemed acceptable.

13.4 Specimen preparation

13.4.1 *Etched specimens*

Specimens should be thoroughly cleaned prior to acid etching (**3.1**) and carefully rinsed to prepare the surfaces for testing in a manner that eliminates the effects of machining, grinding or other techniques used to obtain a specimen of the desired site. Any etching technique that produces a desired smooth and shiny finish free of stains may be used. Zirconium-niobium alloys etch to a matt finish. An example of a suitable procedure for etching and rinsing is given in annex A.

13.4.2 *As-manufacturer specimens*

These specimens should be thoroughly cleaned prior to testing to avoid contamination of the autoclave which could, in turn, adversely affect other specimens used in the test. The extent of specimen cleaning shall be specified by agreement between contracting parties. An example of a suitable cleaning procedure is given in annex A.

14 Procedure

14.1 Inspection of specimens

Examine the specimen for folds, cracks, blisters, foreign material, lustre, brown acid stain, etc. Discard or re-prepare any etched specimen exhibiting acid stain or dull surfaces.

14.2 Dimensions, weight and inspection

Measure each test specimen, either before or after testing, to ± 50 µm and calculate the surface area rounded off to the nearest 1 mm^2. Weigh the specimens to the nearest 0,1 mg with the analytical balance (**8.5**). Check the balance with a calibrated mass in the appropriate range before use and zero it after each fourth weighing. Weigh the specimens when they are thoroughly dry and at the same temperature as the balance.

14.3 Autoclaving

14.3.1 *Placing of test specimens*

Place the clean and weighed test specimens on the clean fixtures in a manner which precludes specimen-to-specimen contact. Autoclaved (**8.3**) stainless steel type 300 or type 400 series washers or wire-mesh grids may be used as separators. Immediately before immersing in the autoclave (**8.3**) the fixtures containing the specimens may be rinsed in grade B water (**7.5**). Immediately before the operation, rinse the autoclave twice with grade B water. Place the specimens and fixtures in the useful volume of the cleaned and rinsed autoclave.

14.3.2 *Venting method A*

14.3.2.1 *Water tests*

It is necessary to determine the volume of water required to ensure that the parts being tested are covered with water during the entire test period. The means of achieving this is as follows.

First pre-determine the required water level by filling the autoclave with sufficient water to cover the samples. Measure the volume of water added, V_1. Calculate the volume of water, V_2 to be added so that the specimens are completely immersed in the liquid phase at the test temperature, using the following equation:

$$V_2 = \frac{V_1}{v'} + \frac{V_3 - V_1}{v''}$$

where

V_3	is the free volume of autoclave (volume of autoclave minus volume of samples), which is best measured by filling the autoclave containing samples to the brim with a measured volume of water;
v' and v''	are the specific volumes of water and steam, respectively, at the test temperature, given in Table 1.

Values for other temperatures are given in the literature [2].

Clean and thoroughly rinse the autoclave before use. Add grade A water to a 10 % excess above the calculate volume, V_2. Activate the autoclave heating units after the autoclave has been attached and sealed. When the internal temperature reaches about 150 °C, open the vent valve to the atmosphere or the venting manifold and collect the condensate to remove the 10 % excess water previously added. Close the valve or valve manifold then control the autoclave for test requirements of temperature and pressure.

14.3.2.2 *Steam tests*

Place the fixtures and specimens in a clean, thoroughly rinsed autoclave. Add grade A water (**7.4**) until the autoclave is at least one quarter to three quarters full. Activate the autoclave heating units after the autoclave has been sealed. When the internal temperature exceeds 110 °C, open the vent valve to the atmosphere or the venting manifold for sufficient time for degassing to be complete, and close the valve. As the temperature and pressure continue to rise, open and close the vent valve, as necessary, to maintain the required pressure.

14.3.3 *Venting method B*

14.3.3.1 *Water tests*

Load the clean, thoroughly rinsed autoclave with fixtures and specimens. Fill with grade A water as in venting method A, or if the autoclave is equipped with an automatic venting system, fill with enough water to cover the specimens. Seal the autoclave and activate the heating units. Vent the autoclave for at least 6 min after 90 °C is reached, then control the autoclave for test requirements of temperature and pressure.

14.3.3.2 *Steam tests*

Load the autoclave with fixtures, specimens and grade A water as in venting method A. Seal the autoclave and activate the heating units. The vent valve shall be open a minimum of 10 min prior to reaching 150 °C. As the temperature and pressure continue to rise, maintain the pressure at test requirements by momentarily opening the vent valve.

14.3.4 *Closed system method C*

14.3.4.1 *Water tests*

Prepare degassed grade A water and store in a separate closed system. Place the fixtures and specimens in the clean, dry autoclave and assemble except for the filling connection. Evacuate the autoclave to approximately 1,3 kPa pressure, backfill with argon (**7.1**) and re-evacuate. Backfill the autoclave with argon to a gauge pressure of 34 kPa, and add the required amount of water without the 10 % excess of method A to the autoclave through a closed system, then seal the autoclave and activate the heating units. No venting is required.

Table 1

Temperature (°C)	**146**	**260**	**288**	**316**	**343**	**360**
v' (liquid)	1,086 2	1,275 5	1,358 5	1,477	1,667	1,894
v'' (steam)	434,7	42,15	26,84	16,55	10,17	6,943

14.3.4.2 *Steam tests*

Use a procedure similar to that described in **14.3.4.1** to fill the autoclave (one quarter to three quarters). After the heating units are activated, vent to attain the required test pressure. Fill the autoclave in a closed system using degassed water. Test the grade A water for pH, electrical resistivity and oxygen content immediately before filling then record the results.

The stated values of pH and electrical resistivity are to be met after the measured values are corrected to 25 °C.

14.3.5 ***Refreshed autoclaving, method D***

A refreshed autoclave with a high-pressure, constant-volume pump, a preheater with controller and a separate over-temperature device, suitable back-pressure controller and system over-pressure protection is required. Operate the pump at rates that permit proper functioning of the pressure control system and maintain the pH within 0,2 units and the electrical resistivity of the effluent water at 0,4 MΩ cm or more. Introduce the feed water, which is initially degassed to less than 45 pg/g oxygen at the bottom of the autoclave and bleed the effluent from the top.

14.3.5.1 *Water tests*

Place samples in clean autoclaves nearly full of grade A water. Close the autoclave cover and connect the necessary piping and instrument lines. Pump additional grade A water into the autoclaves until no air bubbles come out of the open bleed-off valve. Close the bleed-off valve and set the controller to the operating pressure. When the autoclave is pressurized and the effluent water meets the resistivity, pH and dissolved oxygen requirements, turn on the heaters and bring the vessel to operating temperature. When the test is completed, turn off the heaters, disconnect the feed water and cool the autoclave to less than 100 °C before opening.

14.3.5.2 *Steam tests*

Follow the procedure for the water test. Autoclave blow-down through a bottom connection or dip tube is permitted if post-test water samples are not required.

14.4 Post-test measurements and inspection

Carefully remove the specimen from the fixtures, using clean gloves or forceps to prevent scratches. The specimens may be rinsed in grade B water (**7.5**) or ethanol (**7.6**) and dried. Store the specimens in a clean, dry container at the same temperature as the analytical balance (**8.5**) for at least 1 h before weighing. Weigh the specimens and measure the dimensions, if needed. The specimens should be examined in accordance with **15.2**.

15 Calculation or interpretation of results

15.1 Calculation of mass gain

Calculate and record the increase in specimen mass per unit area using the equation:

$$\Delta m = \frac{(m_2 - m_1)}{A}$$

where

Δm is the mass gain per unit area;

m_1 is the pre-test mass of the specimen;

m_2 is the post-test mass of the specimen;

A is the total surface area of the specimen.

NOTE 2 Generally, throughout industry the mass gain is reported in milligrams per square decimeter (mg/dm^2).

15.2 Visual interpretation of surface appearance

After testing examine each specimen for colour, lustre, surface irregularities and corrosion products, and compare against visual standards and record the results. The visual examination shall be performed in a light environment as agreed upon between the purchaser and the testing laboratory.

15.3 Invalid tests

Any test not meeting the parameters of **12.3.3**, **13.1**, **13.2** and **13.3** or where the test operator can define a condition that is significantly different from that normally observed may be declared invalid and the test repeated. The repeated test and the reason for it shall be cited in the test report (clause **16**).

16 Test report

The test report shall include the following information:

a) reference to this International Standard;

b) laboratory where testing was performed;

c) autoclave number and test date;

d) tested material description (type of alloy, chemical composition, lot number, heat treatment, surface finish, etc.);

e) electrical resistivity and pH value (if measured) of water before test;

f) test temperature, pressure, time, type of test and venting method;

g) mass gain, when required and remarks on the visual appearance of each specimen and control coupon;

h) reference to criteria for visual appearance of specimens and control coupons.

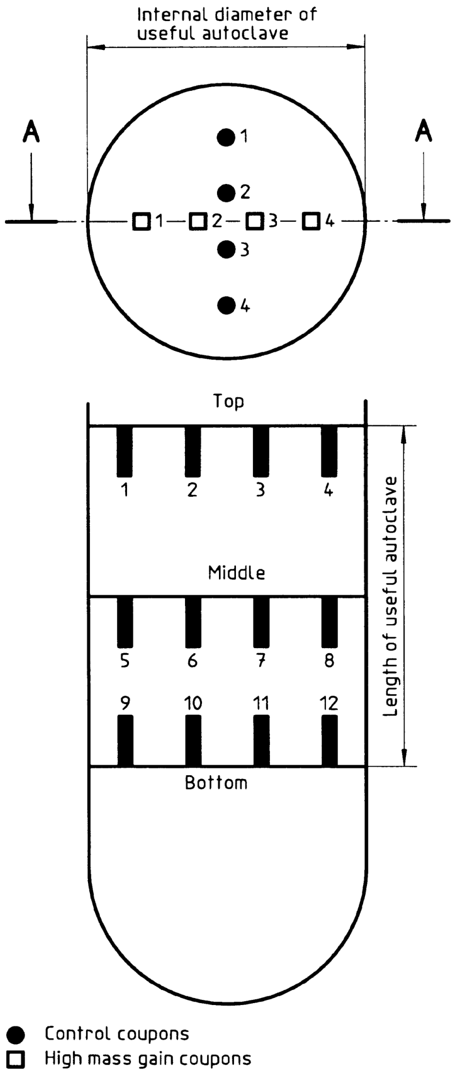

Figure 1 — Control coupon and high mass gain coupon positioning for establishing mass gain mean and standard deviation

Annex A (informative) Guide to specimen preparation

A.1 Tubes with a second material clad on inner diameter

When it is necessary to corrosion test such tubes, the inner surface cladding should be completely removed to avoid erroneous results due to differences in corrosion rates for the two materials.

A.2 Cleaning

Clean the specimens with chemical detergents or organic solvents (**7.6**). A non-metallic brush may be used if required. If solvents are used, the specimens may be cleaned by wiping or total immersion. Immediately after detergent cleaning, thoroughly rinse the test specimens in hot (about 50 °C) flowing water for at least 5 min. After cleaning, handle all specimens with clean, lint-free gloves. Clean the surfaces of all tools, fixtures and the like that come into contact with the clean specimens in a manner equivalent to that used for the specimens. Store all specimens so as to maintain cleanliness.

A.3 Etching

A.3.1 The approximate bath composition for unalloyed zirconium and the zirconium-tin alloy is 3 % (*m*/*m*) of hydrofluoric acid (**7.7**), 39 % (*m*/*m*) of nitric acid (**7.8**) and the remainder distilled or demineralized water. Other concentrations of reagents may be used provided the equivalent final concentrations be obtained. The etching bath temperature should not exceed 50 °C.

A.3.2 For zirconium-niobium alloys, the bath composition is 9 % (*m*/*m*) ± 1 % (*m*/*m*) of hydrofluoric acid, 30 % (*m*/*m*) ± 5 % (*m*/*m*) of nitric, 30 % (*m*/*m*) ± 5 % (*m*/*m*) of sulfuric acid (**7.9**) and the remainder distilled or deionized water. This bath should be controlled between 50 °C and 60 °C.

A.3.3 Generally, 50 µm – 100 µm of the surface of each coupon is removed by etching. Since the rate of metal dissolution is a function of both temperature and acid concentration, the etching rate is determined with a special test coupon before actual test specimens are etched. The etching rate should be checked periodically when a large number of specimens are etched. For the zirconium-tin alloys, the batch should be discarded when the etching rate is less than 25 µm/min per surface or when a total of 425 cm^2 of surface area per litre of acid has been etched.

A.3.3.1 When etching zirconium-niobium alloys it is necessary to limit the area of specimens etched at one time to 30 cm^2 per litre of solution to get a good surface finish, but the bath need not be discarded each time.

A.3.3.2 Freshly etched zirconium alloy surfaces should be bright and lustrous and the bath should not cause preferential attack, except at the top edge or around the holes and identification marks. If preferential etching does occur elsewhere, the test specimen should be discarded or abraded and re-etched. If staining does occur the specimen should be re-etched.

A.3.4 Load the test specimens on to the etching fixture and transfer to the etching bath. (Ensure that the bath temperature is within the limits set above and that only the test specimens and hooks are immersed.) Either

— completely withdraw and alternately immerse the specimens in the etching fixture in the bath at a rate of 60 cycles per minute minimum (a cycle is defined as one immersion and withdrawal)

or

— completely immerse the specimens on the etching fixture in the bath and agitate vigorously. The agitation may be accomplished by bubbling air into the etching station.

A.4 Preliminary rinse

A.4.1 Test specimens should be transferred from the etching bath to the rinse solution as rapidly as possible to prevent staining by the acids. If any acid product remains on the surface or is allowed to dry on the surface, the specimens will not rinse clean and must be re-etched.

A.4.2 After etching is completed, immediately transfer the fixture and test specimens to the first rinse tank, which contains cool (25 °C maximum) flowing water and completely immerse for at least 5 min. The flow rate of the first rinse should be at least two bath changes per minute. A bath change is defined as the flow rate, litres per minute per tank capacity in litres. If the rinse becomes cloudy, allow the specimens to remain completely immersed until the effluent water is clear.

If the local tap water supply is excessively hard, it may be difficult to prevent precipitation of fluorides. In such cases, preliminary rinses may be performed in flowing distilled or deionized water or in a 25 % (*V*/*V*) nitric acid solution at room temperature. The preliminary rinsing of zirconium-niobium alloys must be in a 50 % (*V*/*V*) nitric acid solution at room temperature as an aid in removal of a black surface residue (smut) which may develop during etching. Following the preliminary rinses with nitric acid, rinse the specimens in running water.

If desired, the test specimens can be treated in a sodium hydroxide-water solution (concentration = 500 g/l, temperature = 80 °C – 90 °C, time = 5 min) in order to obtain low fluorine ion concentration ($< 0{,}2\ \mu g/cm^2$) on their surface after chemical etching.

A.5 Final rinse

A.5.1 General

The final rinse is in grade B water (**7.5**). The final rinse may be performed in either a dynamic or a static system. Post-rinsing inspection will not always indicate faulty rinsing operations. Improper techniques will be dramatically observed at the conclusion of the corrosion test as erroneous mass gains and white or generally cloudy surfaces.

A.5.2 Dynamic system

Completely immerse the fixture and test specimens in the rinse tank which contains hot (79 °C minimum) grade B water. Monitor the outlet water for purity. Allow the specimens to remain in the rinse long enough for the effluent water to reach an electrical resistivity of 0,1 MΩ·cm.

A.5.3 Static system

Rinse in running hot water and then completely immerse the test specimens for 5 min in a tank containing hot (91 °C minimum) grade B water. Change the bath whenever the electrical resistivity drops below 0,1 MΩ·cm at room temperature.

A.6 Drying

Cleaned or etched test specimens may be air-dried, wiped dry with a clean, lint-free cloth or blown free of moisture with dry air that is free from dust and acid fumes; any of these methods may be used with or without a prior immersion in ethanol (**7.6**). Handle cleaned and etched specimens only with forceps or clean, lint-free gloves. Keep the test specimens in a clean container when they are not being processed.

Annex B (informative) Bibliography

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

[2] VOUKALOVITCH, M.P., *Thermodynamic Properties of Water and Steam,* Verlag Technik, Berlin.

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