



# Standard Test Method for Corrosion Testing of Products of Zirconium, Hafnium, and Their Alloys in Water at 633°K or in Steam at 673°K [Metric]<sup>1</sup>

This standard is issued under the fixed designation G 2M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

<sup>ε1</sup> NOTE—Section 18 was added editorially in April 1996.

## 1. Scope

1.1 This test method covers (1) the determination of mass gain, and (2) the surface inspection of products of zirconium, hafnium, and their alloys when corrosion tested in water at 633°K or in steam at 673°K.

1.2 This test method is to be utilized in its entirety to the extent specified herein as a product acceptance test.

1.3 This test method may be used on wrought products, castings, powder metallurgy products, and weld metals.

1.4 This specification is the metric companion of Test Method G 2.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 9.

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 888 Test Methods for Dissolved Oxygen in Water<sup>2</sup>

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications<sup>3</sup>

## 3. Terminology

### 3.1 Definitions of Terms Specific to This Standard:

3.1.1 *control coupons*—zirconium alloy specimens of known performance used to monitor the validity of the test.

3.1.2 *etching*—a process for removal of surface metal by action of acids in water.

3.1.3 *Grade A Water*—purified water having a pH of 5.0 to 8.0 and an electrical resistivity of not less than 1.0 M Ω·cm.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee G-1 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.08 on Corrosion of Nuclear Materials.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 14.02.

3.1.4 *Grade B Water*—water prepared with deionized or demineralized water having a minimum electrical resistivity of 0.5 M Ω·cm.

3.1.5 The stated values of pH and electrical resistivity are to be met after the measured values are corrected to 298°K.

3.1.6 *high mass gain coupons*—zirconium alloy specimens that have been specially heat-treated to produce a mass gain higher than the maximum specified as acceptable value used for verifying the severity of the test.

3.1.7 *reagent grade*—the grade of chemicals normally used for analytical purposes.

## 4. Summary of Test Method

4.1 Specimens of zirconium, hafnium, or their alloys are exposed to high-pressure water or steam at elevated temperatures for 72 or 336 h. The corrosion is normally measured by the gain in mass of the specimens and by the appearance of the oxide film on the specimen surfaces. In some instances, such as weld evaluation, mass gain measurements are either impractical to make or not required. When so specified, appearance of the specimen shall be the sole criterion for acceptance.

## 5. Significance and Use

5.1 This test method is primarily used as an acceptance test for products of zirconium, hafnium, and their alloys. This standard has been widely used in the development of new alloys, heat treating practices, and for evaluation of welding techniques.

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

5.3 When tubing with a second material clad on the inner surface is to be tested, the inner cladding shall be removed prior to test.

## 6. Interferences

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

## 7. Apparatus

7.1 The apparatus consists of equipment for (1) etching the specimens when required, (2) measuring the specimen surface area and mass, the water resistivity and pH, test temperature and pressure, etch and rinse temperature, and (3) performing the water or steam corrosion test at elevated temperature and pressure.

7.1.1 *Etching*—An acid bath, a flowing rinse, and a deionized water rinse are needed for proper metal removal and stain-free rinsing. Polyethylene or polypropylene tanks are commonly used with a bottom feed for flowing 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.

7.1.2 *Autoclaves*—The autoclaves are constructed of Type 300 series stainless steel or nickel-base alloys such as UNS grade N06600 or N06690 and are manufactured to conform to ASME and 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 adequate to meet the requirements of this standard are needed. Sample holders and other internal accessories are also constructed of Type 300 or 400 series stainless steel, or nickel-base alloys such as UNS grade N06600 or N06690.

NOTE 1—If autoclave heating is performed in an oven, the oven and not the autoclave will have the automatic temperature-control equipment.

7.1.3 *Measuring Equipment*, capable of measuring specimen dimensions to  $5 \times 10^{-5}$  m and a balance capable of weighing specimens to  $1 \times 10^{-4}$  g are needed.

## 8. Reagents and Materials

8.1 *Argon Gas*, welding grade.

8.2 *Grade A Water*.

8.3 *Grade B Water*.

8.4 *Detergents and Solvents*, for specimen cleaning including reagent grade ethanol and reagent grade acetone.

8.5 *Hydrofluoric Acid (HF)*, reagent grade.

8.6 *Nitric Acid (HNO<sub>3</sub>)*, reagent grade.

8.7 *Sulfuric Acid (H<sub>2</sub>SO<sub>4</sub>)*, reagent grade.

8.8 *Nitrogen Gas*, for purging or controlling oxygen content.

8.9 *Argon-Hydrogen Mixed Gas*, for purging or controlling oxygen content.

## 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, hafnium, and their 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 flammables.

9.4 Cold water should not be added directly to the autoclave

vessel in order to accelerate cooling upon 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.

10.2 Each specimen and control coupons shall be individually identified.

## 11. Preparation of Apparatus

11.1 General requirements for new or reworked autoclaves and parts of autoclaves previously used for testing materials other than to this standard are as follows:

11.1.1 Before specimens are tested in a new or reworked autoclave, or in one having new valves, tubing, gaskets, etc., which contact the test specimen, clean the apparatus thoroughly, wipe with reagent grade ethanol or acetone, and rinse twice with Grade B water. 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, and 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, rinse in hot Grade B water. Autoclave the mixtures and jigs for at least 1 day at 673°K in 10.3 MPa steam or at 633°K in water. Inspect the parts for corrosion product. If corrosion product is found or electrical resistivity of the residual water after the test measures less than 0.1 M Ω-cm, the parts should be cleaned and autoclaved again.

11.2 General requirements for autoclaves and parts in continuous use for corrosion testing under this standard are as follows:

11.2.1 With Grade B water rinse all autoclaves, fixtures, parts, and jigs that have been in continuous use and have shown satisfactory behavior in prior tests. Inspect the fixtures and jigs for corrosion products after each test and rework and re-prepare items showing loose corrosion product.

## 12. Calibration and Standardization

12.1 *High Mass Gain Coupon Preparation*—These coupons shall be selected from a previously tested lot. The selected material shall be heat treated to produce the desired mass gain. Heating for 8 h at 1173°K  $\pm$  3°K and cooling to 573°K  $\pm$  3°K at a rate not exceeding 3.3°K/min will normally produce the desired mass gain.

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 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.

12.2.2 *Establishing Mass Gain Mean and Standard Deviation of Control Coupons*—The control coupon lot and, when

desired, the high mass gain coupon, lot mass gain mean, and standard deviation shall be established by a minimum of one autoclave test as follows:

12.2.2.1 Randomly select 12 specimens from the control coupon lot or the high mass gain coupon lot respectively.

12.2.2.2 Prepare all specimens per the pretest requirements of this test method.

12.2.2.3 Locate the 12 or 24 specimens in a fixture or jig, per Fig. 1, and place the fixture or jig inside the useful volume of the autoclave.

12.2.2.4 Complete the steam or water corrosion test per any one of the four methods in 14.3.

12.2.2.5 Remove specimens and weigh per the requirement of this test method.

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

12.2.2.7 For the product acceptance tests the mean value and standard deviation for the control coupons may be the value established in 12.2.2.6 or may be calculated periodically using all accepted values determined over the preceding 3-month period but not less than 21 values.

12.2.3 An alternate method for establishing the mass gain mean and standard deviation for the control coupons which are used repeatedly is:

12.2.3.1 Expose the control coupons to be used in three different tests, once each in the top, middle, and bottom of an autoclave, and determine mass gain.

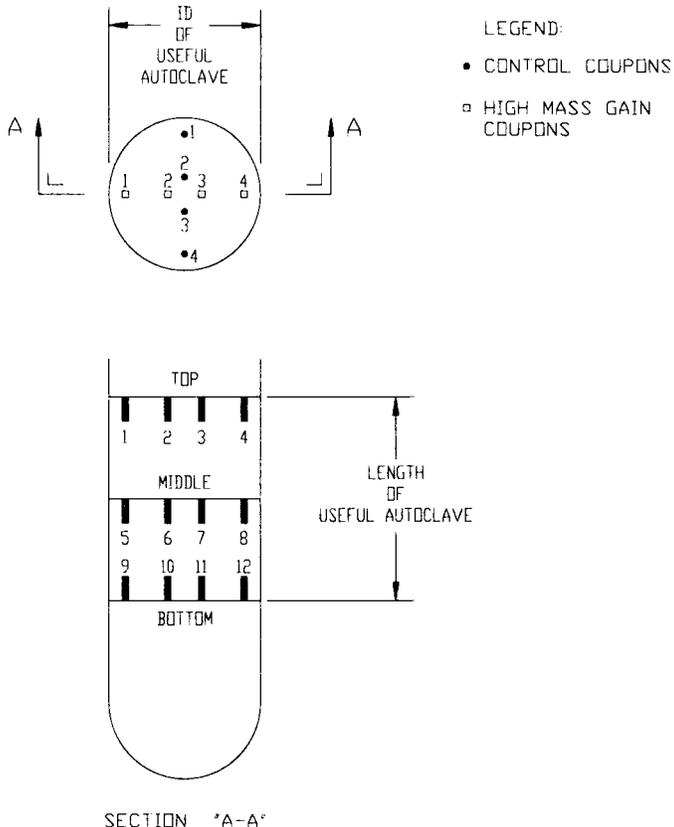


FIG. 1 Control Coupon and High Mass Gain Coupon Positioning for Establishing Mass Gain Mean and Standard Deviation

12.2.3.2 The mean value of each control coupon is the mean for the three tests.

12.2.3.3 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.

12.2.4 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  $\pm 3$  standard deviations.

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 should exhibit no stains, pits, or areas of abnormal etching attack.

12.3.3 An autoclave test is considered acceptable only if each post-test control coupon mass gain is not less than the established mean value minus 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 plus 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 reused after removal of oxide film.

12.4 Calibration:

12.4.1 The temperature measurement and recording systems used to determine conformance shall be calibrated at least every 6 months and shall not deviate more than  $\pm 2^\circ\text{K}$  from the calibration standards traceable to NBS or other known national standards.

12.4.2 Vertical thermal profiles of the autoclaves at the test temperatures shall be made at least once in each 6-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  $\pm 2.2^\circ\text{K}$  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 center 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  $\pm 0.35$  MPa of the calibration standard 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 and have an oxygen content not exceeding 45 ppb. The oxygen content specification may be met either by direct measurement using the appropriate method in Test Method D 888 or by the use of Venting Method A or B of 14.3.

13.2 Autoclave Load Restrictions—The surface area of specimens loaded in a static autoclave shall not exceed 0.1

m<sup>2</sup>/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  $\pm 3^{\circ}\text{K}$  for steam tests and  $\pm 6^{\circ}\text{K}$  for water tests.

13.3.2 *Pressure*—The recorded pressure shall be as specified  $\pm 0.7$  MPa for steam tests and  $\pm 1.4$  for water tests.

13.3.3 *Time*—The exposure time tolerance at the specified temperature and pressure shall be +8 h, -0 h. The time at specified conditions need not be continuous.

13.3.4 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 behavior, may at the option of the test laboratory be deemed acceptable.

### 13.4 Specimen Preparation:

13.4.1 *Etched Specimen*—Specimens should be thoroughly cleaned prior to acid etching and careful rinsing 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 size. Any technique that produces the desired smooth and shiny finish free of stains may be used. Zirconium-niobium alloys etch to a matte finish. An example of a suitable procedure for etching and rinsing is given in Appendix X1.

13.4.2 *As-Manufactured Specimens*—These specimens should be thoroughly cleaned prior to testing to avoid contamination of the autoclave which could adversely affect other specimens in the test. The extent of the specimen cleaning is often specified by agreement between contracting parties. The cleaning section of Appendix X1 is an example of suitable cleaning procedure.

## 14. Procedure

14.1 *Inspection of Specimens*—Examine the specimens for folds, cracks, blisters, foreign material, luster, brown acid stain, etc. Discard or re-prepare any etched specimen exhibiting the acid stain or dull surfaces.

14.2 *Dimensions, Weight, and Inspection*—Measure each test specimen, either before or after testing, to  $\pm 5 \times 10^{-5}$  m and calculate the surface area rounded off to the nearest  $1 \times 10^{-6}$  m<sup>2</sup> in accordance with Practice E 29. Weigh the specimens to the nearest  $1 \times 10^{-4}$  g with an analytical balance checked daily with a calibrated mass before use and zeroed before each fifth weighing. Do not weigh specimens until they are thoroughly dry and at the same temperature as the balance.

### 14.3 Autoclaving:

14.3.1 Place the clean and weighed test specimens on the clean fixtures in a manner precluding specimen-to-specimen contact. Corrosion-film stainless steel Type 300 or 400 series washers or wire mesh grids may be used as separators. Immediately before immersing in the autoclave, the fixtures containing the specimens may be rinsed in Grade B water. Immediately before 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*—Fill the clean, thoroughly rinsed autoclave with enough Grade A water to cover the parts being tested during the entire test period. Calculate the amount of

water to be added so that the specimens are completely immersed in the liquid phase at the test temperature as follows:

$$\text{Volume of water required, } L = k(V_0 - V_1) \quad (1)$$

where:

$V_0$  = autoclave volume,  $L$

$V_1$  = total volume in  $L$  of specimens and fixture and is calculated by dividing the total mass in grams of specimens and fixture by 7 000.

$k$  = 0.8348 at 419°K,  
0.6329 at 533°K,  
0.5954 at 561°K,  
0.5550 at 589°K,  
0.4980 at 616°K, and  
0.4489 at 633°K.

Add a 10 % excess of water. Activate the autoclave heating units after the autoclave has been attached and sealed. When the internal temperature reaches about 420°K, open the vent valve to the atmosphere or the venting manifold as necessary for sufficient time for degassing to be complete, but not to uncover the specimens and 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 until the autoclave is one-fourth to three-fourths full. Activate the autoclave heating units after the autoclave has been sealed. When the internal temperature has exceeded 383°K, 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 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 366°K is reached. Then control the autoclave for the 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 422°K. 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 10 mm Hg pressure, backfill with argon, and re-evacuate. Backfill the autoclave with argon to a gage 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.

14.3.4.2 *Steam Tests*—Use a procedure similar to that described in 14.3.4.1 to fill the autoclave (one-fourth to three-fourths full). After the heating units are activated, vent to attain the required test pressure. The autoclave is filled in a closed system using degassed water. Test the Grade A water for pH, conductivity, and oxygen content immediately before filling and then record the results.

**TABLE 1 Mass Gain Results of Repeated Tests in 673°K Steam<sup>A</sup>**

Laboratory	Mass Gain in g/m <sup>2</sup>					
	3-Day Test				14-Day Test	
	Etched Specimens		As-Manufactured Specimens		Etched Specimens	
	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation
A	1.39	0.174	1.67	0.119	...	...
B	1.47	0.110	...	...	2.62	0.15
C <sup>1,A,B</sup>	1.65	0.106	1.71	0.057	...	...
C2	1.60	0.142	1.60	0.047	...	...
C3	1.69	0.147	1.64	0.087	...	...
C4	1.61	0.074	...	...	...	...
D	1.40	0.15	...	...	...	...

<sup>A</sup> One laboratory reported data on the High Mass Gain Coupons in both the 3- and 14-day tests in 673°K steam (see Table 2).

<sup>B</sup> Laboratory C reported data for each of four autoclaves. Other laboratories pooled data from all of their autoclaves.

14.3.5 *Refreshed Autoclaving, Method D*—A refreshed autoclave with a high-pressure, constant-volume pump, a pre-heater with controller and 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 M Ω-cm or more. Introduce the feed water, which is initially degassed to less than 45 ppb 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 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 373°K before opening.

14.3.5.2 *Steam Tests*—Follow the procedure for 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 or reagent grade ethanol and dried. Then store the specimens in a clean, dry container at the same temperature as the balance for at least 1 h before weighing. Weigh the specimens and measure dimensions, if needed.

**TABLE 2 High Weight Gain Coupons Data in 3- and 14-Day Tests in 673°K Steam**

Test	Mass Gain in g/m <sup>2</sup>	
	Mean Value	Standard Deviation
3 day	6.56	0.81
14 day	8.01	0.76

**15. Calculation or Interpretation of Results**

15.1 *Calculation of Mass Gain*—Calculate and record the increase in specimen mass using the equation:

$$\Delta W = \frac{(W_2 - W_1)}{A} \tag{2}$$

where:

- $\Delta W$  = mass gain, g/m<sup>2</sup>
- $W_1$  = pre-test mass of the specimen, g
- $W_2$  = post-test mass of the specimen, g
- $A^2$  = total surface area of the specimen, m<sup>2</sup>

NOTE 2—Generally, throughout the industry the mass gain is reported in mg/dm<sup>2</sup>.

15.2 *Visual Interpretation of Surfaces:*

15.2.1 *Post-Test Specimen Visual Appearance*—Examine each specimen for color, luster, surface irregularities, corrosion products, and compare against visual standards and record 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 report, Section 16.

**16. Report**

16.1 Record in the laboratory records the following information:

- 16.1.1 Laboratory where test is performed.
- 16.1.2 Autoclave number and test date.
- 16.1.3 pH and resistivity of water before test.
- 16.1.4 Test temperature, pressure, time, type of test, and autoclaving method.
- 16.1.5 Mass gain, when required, and visual appearance remarks of each specimen and control coupon.
- 16.1.6 Comparative criteria for visual appearance of specimens and control coupons.

**17. Precision and Bias**

17.1 *Precision:*

17.1.1 *Statement on Reproducibility*—An industry round-robin was conducted and reported in ASTM STP 458, pages 360–371. The interlaboratory test series using the 14-day, 673°K, 10.3 MPa steam test on a single lot of Grade R60802 Zircaloy yielded an average mass gain of 2.82 g/m<sup>2</sup> with a standard deviation of 0.33 g/m<sup>2</sup>. Similar round-robins have not been run on other variations of time and temperature conditions listed in Test Method G 2.

17.1.2 *Statement on Repeatability*—Table 1, Table 2 tabulates the four laboratories reported data from at least 20

consecutive autoclave tests at 673°K in steam. Each laboratory used its own test lot for the 20 consecutive tests. Three specimens were exposed in each test. The as-manufactured specimens were tubing with a belt-ground outer surface and an inner surface that was either etched or blast abraded. Data on the 633°K water test were not available in sufficient quantity to present.

17.2 *Bias*—Since there is no accepted reference material suitable for determining bias for the procedures in Test Method G 2 for corrosion mass gain, no statement on bias is being made.

## 18. Keywords

18.1 mass gain; steam corrosion; water corrosion

## APPENDIXES

### (Nonmandatory Information)

#### X1. GUIDE TO SPECIMEN PREPARATION

X1.1 *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 difference in corrosion rates of the two materials.

X1.2 *Cleaning*—Clean the specimens with chemical detergents or organic solvents. A nonmetallic 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 322°K 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 in contact with the clean specimens in a manner equivalent to that used for the specimens. Store all specimens so as to maintain cleanliness.

#### X1.3 *Etching:*

##### X1.3.1 *General*

The approximate bath composition for unalloyed zirconium and the zirconium-tin alloys is: 3 mass % of hydrofluoric acid, 39 mass % of nitric acid, and the remainder distilled or demineralized water. Other concentrations of reagents may be used provided the equivalent final concentrations are obtained. The etching bath temperature should not exceed 322°K.

X1.3.2 For zirconium-niobium alloys, the bath composition is  $9 \pm 1$  % of hydrofluoric acid,  $30 \pm 5$  % of nitric acid,  $30 \pm 5$  % of sulfuric acid, and the remainder distilled or deionized water. This bath should be controlled to 322°K to 333°K.

X1.3.3 Generally,  $1 \times 10^{-5}$  to  $1 \times 10^{-4}$  m 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 tests specimens are etched. The etching rate should be checked periodically when a large number of specimens are etched. For the zirconium-tin alloys, the bath should be discarded when the etching rate is less than  $2.5 \times 10^{-5}$  m/min per surface or when a total of  $4.25 \times 10^{-2}$  m<sup>2</sup> of surface area per litre of acid has been etched.

X1.3.3.1 When etching the zirconium-niobium alloys, it is necessary to limit the area of specimens etched at one time to  $3 \times 10^{-3}$  m<sup>2</sup>/L of solution to get good surface finish, but the bath need not be discarded each time.

X1.3.3.2 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.

X1.3.4 Load the test specimens on the etching fixture and transfer to the etching bath. (Assure that the bath temperature is within the limits set forth above and that only the test specimens and hooks are immersed.) Either (a) completely withdraw and immerse alternately the specimens in etching fixture in the bath at a rate of 60 cpm minimum (a cycle is defined as one immersion and withdrawal) or (b) 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. Limit the total etching time to that required to remove  $1 \times 10^{-5}$  to  $1 \times 10^{-4}$  m per surface, as determined above with a test coupon.

#### X1.4 *Preliminary Rinse:*

##### X1.4.1 *General*

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.

X1.4.2 After the etching is completed, immediately transfer the fixture and test specimens to the first rinse tank, which contains cool (298°K maximum) flowing water and completely immerse for at least 5 min. The flow rate of the first rinse should be at least 2 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.

X1.4.2.1 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 % HNO<sub>3</sub> solution at room temperature. The preliminary rinsing of zirconium-niobium alloys must be in a 50 volume % HNO<sub>3</sub> 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 HNO<sub>3</sub>, rinse the specimens in flowing water.

### X1.5 Final Rinse:

#### X1.5.1 General

The final rinse is in Grade B water. 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.

#### X1.5.2 Dynamic System

Completely immerse the fixture and test specimens in the rinse tank which contains 352°K 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 M Ω-cm, plus 5 min.

### X1.5.3 Static System

Rinse in flowing hot tap water and then completely immerse the test specimens for 5 min in a tank containing (364°K minimum) Grade B water. Change the bath whenever the electrical resistivity drops below 0.1 M Ω-cm.

X1.6 *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 of dust and acid fumes; any of these methods may be used with or without a prior immersion in reagent grade alcohol. 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.

## X2. RATIONALE (COMMENTARY) ON REVISION OF ASTM G 2M – 81

X2.1 The ASTM rules require a standard to be reviewed every five years for adequacy of technical contents. This standard, ASTM G 2M – 81, was reviewed in 1986 and incorporates the following:

X2.1.1 Title was changed from Standard Practice to Standard Test Method since this standard is used throughout the industry as a product acceptance test. In addition the title was revised to clarify the two distinct test conditions; one for 633°K water and the other for 673°K steam. The word “hafnium” was also added to the title.

X2.1.2 Over the years this standard has undergone numerous changes as well as form and style of ASTM standards. This revision has been restructured to follow guidelines for ASTM test methods given in *Form and Style for ASTM Standards*, 7th Edition, March 1986.

X2.1.3 Additional definitions have been added to eliminate confusion regarding terminologies such as control coupons and high mass gain coupons.

X2.1.4 The common terminology for reporting of data is weight gain in mg/dm<sup>2</sup>. However, to conform to the SI terminology, the unit of area is given in m<sup>2</sup>, and terms “weight” and “weight gain” have been replaced with “mass” and “mass gain” since the unit of measure is in grams.

X2.1.5 The acceptance requirements for an autoclave test have been revised to be based on control coupons mass gain standard deviations from the mean value rather than an arbitrary value previously used. This change was incorporated in order to establish a more rigorous acceptance criteria and to control the autoclave test better within a run and between runs.

X2.1.6 A method for establishing the mean and standard

deviation of control coupons has been added in this revision to standardize the procedure. An alternate method for control coupons which are used repeatedly is also added.

X2.1.7 Requirements for reporting results were established in this revision.

X2.1.8 The detailed sample preparation section has been moved to a non-mandatory guide in the appendix because of wide variety of techniques used in the laboratories.

X2.1.9 There remain variables affecting this test that are as yet unidentified that may lead to excessive corrosion or lack of corrosion. It is suspected that one of these variables is the presence of impurities in the water at concentration not detectable at this time. The control scheme to compensate for this feature is the presence of control coupons in the test on which validity or severity of the test is measured. This control is imperfect because of variability in performance among specimens in any lot of control coupons.

X2.1.10 High mass gain coupons have been used to assure minimum test sensitivity by some laboratories. This practice is encouraged to form a data base consideration of the high mass gain coupons as a mandatory requirement in future revisions of this standard.

X2.1.11 In steam test by venting method A, degassing of steam results in reducing the oxygen content of the steam to a minimum level. However, the venting time required to achieve the desired oxygen level is dependent upon the initial amount of water, autoclave pressure, and temperature during venting and venting duration and frequency. Therefore it is impractical to specify a minimum venting time.

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