



Designation: ~~G2/G2M-06 (Reapproved 2011)~~<sup>ε1</sup> G2/G2M-19

## Standard Test Method for Corrosion Testing of Products of Zirconium, Hafnium, and Their Alloys in Water at 680°F (360°C) or in Steam at 750°F (400°C)<sup>1</sup>

This standard is issued under the fixed designation G2/G2M; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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<sup>ε1</sup> NOTE—Editorial corrections were made in Section 14.3.4.1 in October 2013.

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### 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 680°F [360°C] or in steam at 750°F [400°C].

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 Unless a single unit is used, for example corrosion mass gain in mg/dm<sup>2</sup>, the values stated in either inch-pound or SI units are to be regarded separately as standard. The values stated in each system are not exact equivalents; therefore each system must be used independently of the other. SI values cannot be mixed with inch-pound values.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements, see Section 9.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D888 Test Methods for Dissolved Oxygen in Water

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

### 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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

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

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

3.1.4 *Grade B water, n*—water prepared with deionized or demineralized water having a minimum electrical resistivity of 0.5 MΩ·cm.

3.1.5 The stated values of pH and electrical resistivity are to be met after the measured values are corrected to 77°F [25°C].

3.1.6 *high mass gain coupons, n*—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.

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<sup>1</sup> This test method is under the jurisdiction of ASTM Committee B10 on Reactive and Refractory Metals and Alloys and is the direct responsibility of Subcommittee B10.02 on Zirconium and Hafnium.

Current edition approved Sept. 1, 2011; Jan. 1, 2019. Published September 2011; February 2019. Originally approved in 1967. Last previous edition approved in 2006; 2011 as G2/G2M-06; G2/G2M-06 (2011)<sup>ε1</sup>. DOI: 10.1520/G0002-G0002M-06R1HE01-10.1520/G0002-G0002M-19.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For Annual Book of ASTM Standards volume information, refer to the standard's Document Summary page on the ASTM website.

NOTE 1—For the purpose of standardization the term “mass gain” is used, however within industry the terms “mass gain” and “weight gain” are used interchangeably.

3.1.7 *reagent grade, n*—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 the 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*, 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 (American Society for Mechanical Engineers) 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 2—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 0.002 in. [ $5 \times 10^{-5}$  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 Do not add cold water 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 shall 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 fixtures and jigs for at least 1 day at 750°F [400°C] in 1500 psi [10.3 MPa] steam or at 680°F [360°C] 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 MΩ-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 1652 ± 5°F [(900 ± 3°C)] and cooling to 572 ± 5°F [300 ± 3°C] at a rate not exceeding 6°F/min [3.3°C/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, in accordance with 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 in accordance with any one of the four methods in 14.3.

12.2.2.5 Remove specimens and weigh in accordance with 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 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 at least the preceding 3-month period but not less than 21 values.

12.2.3 An alternative 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.

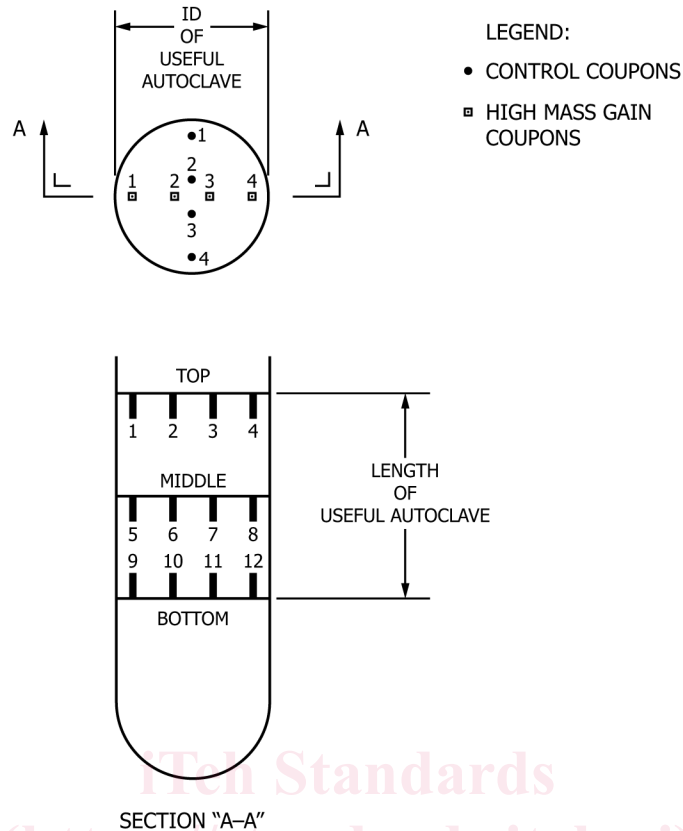


FIG. 1 Control Coupon and High Mass Gain Coupon Positioning for Establishing Mass Gain Mean and Standard Deviation

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 failed 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  $\pm 5^\circ\text{F}$  [ $\pm 3^\circ\text{C}$ ] from 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  $\pm 5^\circ\text{F}$  [ $\pm 3^\circ\text{C}$ ] 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 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  $\pm 50$  psi [ $\pm 0.35\text{MPa}$ ] 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 Methods **D888** 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 ±5°F [±3°C] for steam tests and ±10°F [±6°C] for water tests.

13.3.2 *Pressure*—The recorded pressure shall be as specified ±100 psig [±0.7 MPa] for steam tests and ±200 psig [±1.4 MPa] 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 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 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 a suitable cleaning procedure.

### 14. Procedure

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

14.2 *Dimensions, Weight, Mass, and Inspection*—Measure each test specimen, either before or after testing, to ±0.002 in. [±5 by× 10<sup>-5</sup> m] and calculate the surface area rounded to the nearest 1 by× 10<sup>-6</sup> m<sup>2</sup> in accordance with Practice **E29**. Weigh the specimens to the nearest 1 by× 10<sup>-4</sup> 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 Type 300 or 400 series stainless steel 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 weight in grams of specimens and fixture by 7000.

$V_1$  = total volume in  $L$  of specimens and fixture, and is calculated by dividing the total mass in grams of specimens and fixture by 7000.

$k$  = 0.8348 at 295°F [146°C],  
 0.6329 at 500°F [260°C],  
 0.5954 at 550°F [288°C],  
 0.5550 at 600°F [316°C],  
 0.4980 at 650°F [343°C], and  
 0.4489 at 680°F [360°C].



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 300°F [147°C], 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 230°F [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 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 200°F [93°C] is reached. Continue to 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 300°F [149°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 10 mm Hg pressure, backfill with argon, and re-evacuate. Backfill the autoclave with argon to a gage pressure of 5 psi [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.

14.3.5 *Refreshed Autoclaving, Method D*—A refreshed autoclave with a high-pressure, constant-volume pump, a preheater 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 MΩ-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 212°F [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 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.

## 15. Calculation and Interpretation of Results

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

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

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, and
- $A$  = total surface area of the specimen, m<sup>2</sup>.

NOTE 3—Throughout the industry the mass gain is generally reported in mg/dm<sup>2</sup>.

15.2 *Visual Interpretation of Surfaces:*