

SEMI F120-0922

TEST METHOD FOR THE ELECTROCHEMICAL CRITICAL PITTING VOLTAGE TESTING OF STAINLESS STEEL USED IN CORROSIVE GAS SYSTEMS

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NOTICE: Paragraphs entitled ‘NOTE:’ are not an official part of this Standard or Safety Guideline and are not intended to modify or supersede the official Standard or Safety Guideline. These have been supplied by the global technical committee to enhance the usage of the Standard or Safety Guideline.

1 Purpose

1.1 The purpose of this Test Method is to determine the relative resistance to pitting corrosion of the wetted surfaces of components intended for use in corrosive gas distribution systems for semiconductor manufacturing. This Test Method is an adaptation of ASTM G61¹ and is intended to differentiate among manufacturing processes for stainless steel that can affect the performance of the wetted surfaces.

2 Scope

2.1 This Test Method describes a procedure, based on the electrochemical critical pitting voltage (CPV), which is used to rank the pitting corrosion resistance of wetted surfaces of tubing or test coupons of representative finished surfaces intended for use in corrosive gas systems. Pitting corrosion is believed to be a major corrosion failure mode in semiconductor gas delivery systems, particularly in components and tubing welded and exposed to corrosive gases and moisture.

2.2 This Test Method is an adaptation of ASTM G61. This adaptation describes a method for performing the test on wetted surface sections of finished fittings such as glands, elbows and blocks as well as tube welds to fittings. It is an aqueous immersion method.

2.3 The test method is reproducible and provides a metric (CPV) in addition to a qualitative (visual) evaluation of the location and extent of the pitting.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 To obtain reliable and reproducible results, only analysts familiar with the instrumentation and technique should use this Test Method.

3.2 The test conditions of this Test Method do not simulate actual service in a corrosive gas supply system. Thus, it is possible that the test results do not correlate to relative corrosion resistance in a specific corrosive gas or corrosive gas supply system.

3.3 The CPV is valid in a range from the corrosion potential to 1 Volt as measured against a standard calomel reference electrode.

3.4 As alloy composition and surface parameters can affect the results of the test, all variables other than the one being tested must be fixed during the test in order to obtain reproducible and comparable results.

3.5 Values stated in SI or SI-derived units are to be regarded as standard. No other units of measurement are included in this Test Method.

¹ Adapted from ASTM G61-86(2018) Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Based Alloys, © ASTM International, 100 Barr Harbor Drive, West Conshohocken PA 19428. A copy of the complete standard may be obtained from ASTM, <http://www.astm.org>.

4 Referenced Standards and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI F19 — Specification for the Surface Condition of the Wetted Surfaces of Stainless Steel Components

4.2 ASTM® Standards²

ASTM G3 — Standard Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing

ASTM G15 — Standard Terminology Relating to Corrosion and Corrosion Testing

ASTM G61 — Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel-, or Cobalt-Based Alloys

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

NOTE 1: Unless otherwise stated, the sign conventions used in this Test Method are in agreement with ASTM G3, and the terminology relating to corrosion and corrosion testing is as defined in ASTM G15.

5.1 Abbreviations and Acronyms

5.1.1 *CPV* — critical pitting voltage

5.1.2 *OCP* — open circuit potential

5.2 Definitions

5.2.1 *crevice corrosion* — corrosion in a narrow opening between adjoining pieces or at the edge of a seal or masking paint.

5.2.2 *critical pitting voltage* — the lowest electrochemical potential at which stable propagating pitting occurs on the test surface under the specified test conditions, as indicated by a rapid increase beyond a specified limit of the measured anodic current of the test surface.

5.2.3 *open circuit potential* — the potential between the test specimen and the counter electrode with no electrical connection.

5.2.4 *pitting* — corrosion of a metal surface, confined to a point or small area, that takes the form of cavities.

5.2.5 *saturated calomel electrode* — a standard reference electrode based on the reaction between elemental mercury and mercury chloride.

NOTE 2: ‘Saturated calomel electrode’ is commonly abbreviated as ‘SCE’, although not in this Test Method.

5.2.6 *wetted surface* — a surface of a component in contact with the contained fluids.

5.3 Symbols

5.3.1 *M* — molar concentration

5.3.2 $\mu\text{A}/\text{cm}^2$ — microamperes per square centimeter

6 Summary of Test Method

6.1 The test method measures the CPV by using a potentiodynamic technique and a polarization scan. The test is performed in an electrochemical cell in a 1 M NaCl aqueous electrolyte solution. The test specimen is polarized at a constant rate of anodic potential of 0.167mV/s until pitting occurs. The test is terminated at a corrosion current of 3 mA.

6.2 Pitting is confirmed by visual observation of the specimen after testing. If necessary, the specimen is sectioned before post-test inspection to ensure unobstructed line(s) of sight to the wetted surface(s).

² ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: +1.610.832.9585, Fax: +1.610.832.9555, <http://www.astm.org>. ASTM trademark is owned by ASTM International.

7 Possible Interferences

7.1 Examine the exterior surface of the part after testing to verify that no defect in the protective lacquer has allowed pitting in an area not on the wetted path.

8 Apparatus

8.1 Use a one liter glass vessel, similar to the one shown in Figure 1, having suitable necks or seals to permit the introduction of electrodes, gas inlet and outlet tubes, a thermometer, and a nitrogen diffuser.



#1 A gland is immersed in the test solution, well below the liquid surface.

#2 The gland is supported by the insulated electrical conductor, which leads out to the potentiostat.

#3 A cylindrical graphite electrode is located at the left.

Figure 1
Example of Test Set-Up

8.2 The specimens or parts will generally be from manufactured components such as glands, elbows, tubing, base blocks, and flanges. Welds to these parts can also be tested.

8.3 Use a potentiostat that will maintain an electrode potential within 1 mV of a preset value over a wide range of anodic currents. The potentiostat should have a potential range of -1.0 to $+1.5$ V and an anodic current output range of 1.0nA to 10mA.

8.4 The potential-measuring circuit should have a high input impedance to minimize current drawn from the system during measurements.

8.5 Use a potentiostat that can be programmed to carry out potentiodynamic scans. Plot and record potential and current continuously.

8.6 Use a graphite or platinum counter electrode. The counter electrode should have an area at least twice as large as the wetted area of the test specimen.

8.7 A saturated calomel electrode with a controlled rate of leakage (about 3 $\mu\text{L/h}$) is recommended. This type of electrode is durable, reliable, and commercially available. Precautions should be taken to ensure that it is maintained in the proper condition. The potential of the calomel electrode should be checked at periodic intervals to ensure the accuracy of the electrode.

9 Test Parts

9.1 Test parts are required to be representative of a product or process lot. Sampling shall be per agreement between component supplier and purchaser.

9.2 Sample cleaning and packaging should preferably be done by the component manufacturer. The sample(s) are to be cleaned and packaged per the manufacturer's standard final cleaning and packaging procedures.

9.3 Document all identifying information for inclusion in the test report before removing test parts from their protective packages.

9.4 On removal from the packaging, make an electrical connection to the external surface of the part. Seal the exposed copper wire and the surrounding part surface with epoxy to avoid direct contact with the test solution. (Refer to Figure 2)

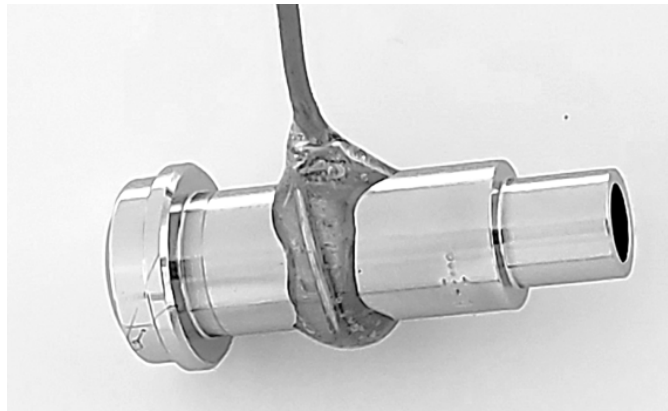


Figure 2
Wired Gland with Epoxy Sealing Exposed Wire Surfaces

9.5 Mask each specimen to isolate the test to the surface of interest only. This can be accomplished by masking with a nonreactive lacquer. (Refer to Figure 3.)