

SEMI F77-0703

TEST METHOD FOR ELECTROCHEMICAL CRITICAL PITTING

TEMPERATURE TESTING OF ALLOY SURFACES USED IN

CORROSIVE GAS SYSTEMS

This test method was technically approved by the Global Gases Committee and is the direct responsibility of the North American Gases Committee. Current edition approved by the North American Regional Standards Committee on April 11, 2003. Initially available at www.semi.org June 2003; to be published July 2003.

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 intended to differentiate between alloy compositions and processes intended to enhance the corrosion performance of the wetted surfaces.

2 Scope

2.1 This test method describes a procedure, based on the electrochemical critical pitting temperature (CPT), 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.

2.2 This test method is an adaptation of ASTM G150. The adaptation describes a method for performing the test method on coupons or wetted-surface sections cut from gas supply system components such as tubing. It is an aqueous immersion method.

2.3 The test method is reproducible and provides a metric (critical pitting temperature) in addition to a qualitative (visual) evaluation of corrosion resistance.

2.4 This test method applies to materials as specified in SEMI Standards referenced in § 4.1, and to welds of these materials.

2.5 This test method may also be used for other corrosion resistant alloys and their welds not referenced in § 4.1.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this standard to establish appropriate safety health practices and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 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 the test results may not correlate to relative corrosion resistance in a specific corrosive gas or corrosive gas supply system.

3.3 The Critical Pitting Temperature (CPT) is valid only in a range of 10°C to 95°C. A CPT result greater than 95°C is not considered to be valid since it is approaching the boiling point of the solution. A critical pitting temperature (CPT) below 10°C shall only be reported as <10°C and may be an indication of crevice corrosion.

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

3.5 The bias for this test has not yet been determined.

4 Referenced Standards and Documents

4.1 SEMI Standards

SEMI F2 — Specification for 316L Stainless Steel Tubing for General Purpose Semiconductor Manufacturing Applications

SEMI F17 — Specification for High Purity Quality Electropolished 316L Stainless Steel Tubing, Component Tube Stubs, and Fittings made from Tubing

SEMI F19 — Specification for the Finish of the Wetted Surfaces of Electropolished 316L Stainless Steel Components

SEMI F20 — Specification for 316L Stainless Steel Bar, Extruded Shapes, Plate, and Investment Castings for Components Used in High Purity Semiconductor Manufacturing Applications

4.2 ASTM Standards¹

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

ASTM G15 — Standard Terminology Relating to Corrosion and Corrosion Testing

ASTM G150 — Test Method for Electrochemical Critical Pitting Temperature Testing of Stainless Steels

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 G15.

5.1 Definitions

5.1.1 *CPT-Critical Pitting Temperature* — the lowest temperature 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 density of the test surface.

5.1.2 *passive potential range* — the potential range over which the current density is independent of potential. The current is a very low value due to formation of an oxide layer.

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

5.1.4 *pitting potential range* — the range of measured potentials where pitting is initiated. This potential range only exists above the minimum CPT.

5.1.5 *potential dependent CPT* — the CPT determined at a potential within the pitting potential range of the tested surface.

5.1.6 *potential independent CPT* — the CPT determined at a potential above the pitting potential range, but below the transpassive potential range.

5.1.7 *temperature ramp* — the rate (°C/min) at which the test temperature is increased during the test.

5.1.8 *transpassive potential* — the potential above the passive potential range, in which the current density increases rapidly as the potential increases.

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

6 Summary of Method

6.1 The test method measures the critical pitting temperature (CPT) by using a potentiostatic technique and a temperature scan. Specific types of specimen holders are used to minimize crevice corrosion, which is another type of corrosion mechanism that can complicate the CPT result. The test is performed in an aqueous electrolyte solution, such as 1 M NaCl. After polarizing the specimen to a potential above the pitting potential, the temperature scan

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begins at 0°C. The solution is heated at a constant rate of 1°C/minute while the current is measured by means of a potentiostat/galvanostat instrument. The CPT, as defined in ¶ 5.1.1, is the temperature at which the current density increases above 100 $\mu\text{A}/\text{cm}^2$ for 60 seconds. Pitting is normally confirmed by visual or microscopic observation of the specimen after testing as shown in Figure 1. Refer to Appendix X1 in ASTM G150 for a description of the relationship between the pitting potential and the critical pitting temperature.

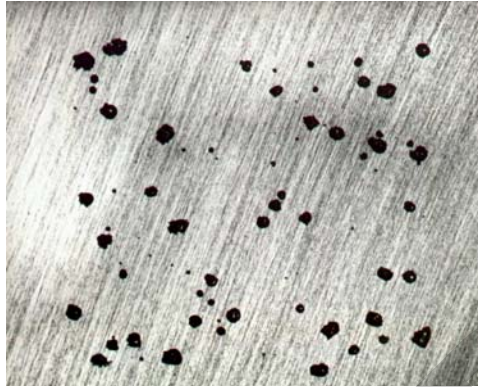


Figure 1
Examples of Pitting on Type 316 Stainless Steel After a
Pitting Potential Test Performed at 25°C in 1 M NaCl (11 × Magnification)^(Ref 17.1)

7 Possible Interferences

7.1 Specimens must be examined after testing to confirm pitting. A possible interference is failure by crevice corrosion in which case the test is invalid. Crevice corrosion can occur at the seal of the specimen holder. Figure 2 shows the mechanism by which crevice corrosion occurs, and Figure 3 gives an example of the typical morphology of crevice corrosion from a pitting potential test performed on type 316 stainless steel using masked edges. It should also be noted that the critical crevice temperature (CCT) for AISI 316L is less than 10°C.

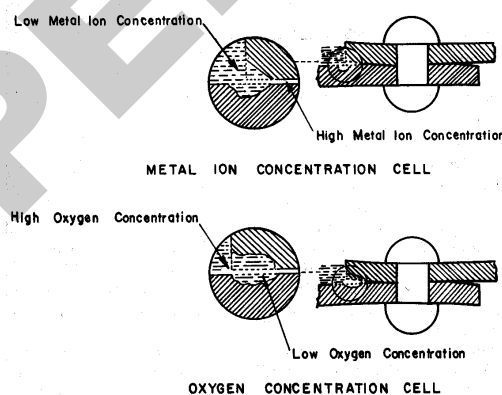


Figure 2
Mechanism for Crevice Corrosion
(See ¶ 17.2.)