

SEMI F77-0915

TEST METHOD FOR ELECTROCHEMICAL CRITICAL PITTING

TEMPERATURE TESTING OF STAINLESS STEEL SURFACES USED IN CORROSIVE GAS SYSTEMS

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NOTICE: This Document was completely rewritten in 2015.

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 G150 and is intended to differentiate between alloy compositions or processes that may affect the corrosion performance of the wetted surfaces.

1.2 Detailed information on the method is found in ASTM G150, and it is recommended that the user have a copy of ASTM G150 for application of this Test Method.

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 and moisture.

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 (CPT) in addition to a qualitative (visual) evaluation of corrosion resistance.

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 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 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 CPT below 10°C shall only be reported as <10°C and may be an indication of crevice corrosion, which renders the test invalid.

3.4 Since 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.

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 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 — Standard 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 ASTM G15.

5.1 Abbreviations and Acronyms

5.1.1 *CPT* — critical pitting temperature

5.1.2 *OCP* — open circuit potential

5.1.3 *SCE* — saturated calomel electrode

5.2 Definitions

5.2.1 *crevice corrosion* — corrosion at a cavity between adjoining pieces or at the edge of a seal or masking paint. The chemical mechanism of crevice corrosion is similar to that of pitting, the cavity being a ‘pre-formed’ pit. As corrosion is initiated at the cavity, the critical temperature for crevice corrosion is always lower than the critical pitting temperature.

5.2.2 *critical pitting temperature (CPT)* — 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.

5.2.3 *flushed port cell* — a type of CPT test cell that utilizes a porous seal against the test specimen through which deionized water is flushed to avoid crevice corrosion at the edge of the seal. This is illustrated and described in ASTM G150. These cells are commercially available.

5.2.4 *Luggin probe* — a capillary ended tube that holds the reference electrode. The capillary end is filled with the test solution and situated close to the test specimen so that the reference electrode senses the solution potential at this open tip. Also known as a Luggin-Haber capillary.

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

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

5.2.7 *SCE reference electrode* — SCE, a standard reference electrode based on the reaction between elemental mercury and mercury chloride.

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

5.3 Symbols

5.3.1 *M* — Molar Concentration

5.3.2 *mV* — Millivolts

5.3.3 $\mu\text{A}/\text{cm}^2$ — Microamperes per square centimeter

6 Summary of Method

6.1 The test method measures the CPT by using a potentiostatic technique and a temperature scan. The test is performed in an electrochemical cell in a 1 M NaCl aqueous electrolyte solution. The test specimen is polarized to a constant anodic potential, typically 700 mV for stainless steel, and the temperature scanned at a rate of 1°C/minute,

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beginning at 0°C. The current is measured by means of a potentiostat/galvanostat instrument. The CPT is the temperature at which the current density increases above 100 $\mu\text{A}/\text{cm}^2$ for 60 seconds. Pitting is confirmed by visual or microscopic observation of the specimen after testing, as shown in Figure 1.



Figure 1
Examples of Pitting on Type 316 Stainless Steel After a
Pitting Potential Test in 1 M NaCl (11× Magnification)

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 considered invalid. Crevice corrosion can occur at the seal of the test specimen to the test apparatus or at the edge of the masking media. The crevice corrosion sites exist at the boundaries of the area exhibiting pitting and can be identified by a straight edge resulting from the masking. Figure 2 gives an example of the typical morphology of crevice corrosion sites from a pitting potential test performed on type 316 stainless steel using masked edges. Crevice corrosion also has a critical temperature which is always less than the CPT. The critical crevice corrosion temperature for AISI 316L is less than 10°C. Test results for AISI 316L less than 10°C achieved by the use of this Test Method are likely due to crevice corrosion and are considered invalid.



Figure 2
Examples of Crevice Corrosion (Dashed Circles) on Type 316 Stainless Steel After a
Pitting Potential Test in 1 M NaCl² (11× Magnification)

² Blum, Michael. M.S. Thesis. University of London, England, December 1970.