

SEMI F74-1103 (Reapproved 0710) TEST METHOD FOR THE PERFORMANCE AND EVALUATION OF METAL SEAL DESIGNS FOR USE IN GAS DELIVERY SYSTEMS

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NOTICE: The section symbol (§) is used to reference a numbered section, paragraph, or header and all subordinate paragraphs, headers, and embedded material (i.e., EXCEPTIONs, lists) therein. The paragraph symbol (§) is used to reference a particular numbered paragraph and embedded material therein. When duplicated (i.e., §§ and §§§) the symbols are used to reference more than one section or paragraph, respectively.

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.

NOTICE: This Standard or Safety Guideline has an Inactive Status because the conditions to maintain Current Status have not been met. Inactive Standards or Safety Guidelines are available from SEMI and continue to be valid for use.

1 Purpose

1.1 This Standard is a test method for evaluating metal seal designs use in gas delivery systems. It covers both surface-mounted gas systems and conventional metal face seal fitting systems.

2 Scope

2.1 The test methods apply to the connection seals used in conventional tubing type gas systems and between modules and components to the substrates used in surface-mounted gas systems.

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 This Test Method does not provide detailed information sufficient for conducting the procedures. It is the responsibility of the user to procure a copy of the referenced test procedures from the issuing organizations.

3.2 The test methods mentioned in this Standard are destructive in some cases. Therefore, the sequence of tests should be carefully planned with the understanding that several specimens are required to complete the battery of all applicable tests without invalidating later tests. This requirement of several specimens is in addition to the iteration normally required under good statistical practices.

3.3 All components must meet quality requirements (dimensional, sealing-surface finish, etc.), as established and controlled by manufacturers prior to testing.

3.4 Surface-mounted connection methods and system design will affect exterior load testing (i.e., vibration, shock, etc.).

3.5 Seals must be manufactured and packaged for Class 100-type applications.

3.6 Care should be exercised in handling seals to maintain manufacturer’s specifications.

4 Referenced Standards and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI F1 — Specification for Leak Integrity of High-Purity Gas Piping Systems and Components

SEMI F77 — Test Method for Electrochemical Critical Pitting Temperature Testing of Alloy Surfaces Used in Corrosive Gas Systems

4.2 ISO® Standards¹

ISO 14644-1 — Cleanrooms and Associated Controlled Environments – Part 1: Classification of Air Cleanliness

4.3 SEMATECH Documents²

SEMASPEC 90120390B-STD — Test Method for Particle Contribution

SEMASPEC 97043272A-TR — Accelerated Life Testing of Gas System Performance and Reliability

4.4 United States Federal Standards³

Federal Standard 209E — Airborne Particle Cleanliness Classes in Cleanrooms and Clean Zones

4.5 United States Military Standards⁴

MIL-STD-810E — Environmental Test Methods and Engineering Guidelines

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

5 Terminology

5.1 Definitions

5.1.1 *cap block* — a seal-specific component fixture specially made to mate with a respective seal-system substrate block for testing purposes.

5.1.2 *Class 100* — a cleanroom designation defined by Federal Standard 209E (ISO 14644-1, -2, -4 equivalent) which designates that each cubic foot (SI equivalent cubic meter) of air can have no more than 100 particles at a size of 0.5 µm or larger.

5.1.3 *component* — an individual piece or a complete assembly of individual pieces capable of being joined with other pieces or components.

5.1.4 *connector block* — a seal-specific substrate block fixture made to mate with a respective seal-system cap or flow-through block for testing purposes.

5.1.5 *conventional system* — a gas system utilizing tubing and standard face seal type weld fittings joined together using orbital TIG welding.

5.1.6 *design pressure* — of a system or subsystem, the pressure at the most severe condition of internal and external pressure for which it is appropriate (as defined by the designer) to use the system or subsystem.

5.1.7 *dummy* — a term meant to denote an imitation of a real or original object. In this case, the original object would be a modular surface mount valve.

5.1.8 *finger-tight* — where a particular joining apparatus (seal-system) is compressed/torqued to the point where one would need a tool of some sort to apply further force.

5.1.9 *fixture* — a device specially designed and manufactured for a particular seal-system and performance test. Sample performance testing fixtures can be seen in Related Information 1.

5.1.10 *flow-through* — a term used to signify that the configuration of a given fixture or substrate must allow for gas to pass through from an inlet interface point to an outlet interface point. A flow-through device allows one to make flow calculations and obtain particle counts.

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² SEMATECH, 257 Fuller Road, Suite 2200, Albany, NY 12203, USA; Telephone: +1.518.649.1000, <http://www.sematech.org>.

³ United States Federal Standards, available as freely downloadable PDF files from the ASSIST Quick Search website at <http://quicksearch.dla.mil>.

⁴ United States Military Standards, available as freely downloadable PDF files from the ASSIST Quick Search website at <http://quicksearch.dla.mil>.

5.1.11 *hydrostatic leak* — a leak or leak test performed by applying isostatic pressure via some sort of liquid phase media (i.e., hydraulic fluid, water).

5.1.12 *inboard leak rate* — leakage rate expressed in $\text{Pa}\cdot\text{m}^3/\text{s}$ ($\text{atm}\cdot\text{cc}/\text{sec.}$) from outside to inside occurring when an internal pressure is less than the external pressure acting on the component or system. Inboard leakage is typically determined by introducing a tracer gas around the exterior of the piping system or component under test.

5.1.13 *leak* — a path (or paths) in a sealed system that will pass tracer gas when a pressure differential or diffusion path exists. There are two leak mechanisms: a mechanical passage and a material through which a gas can diffuse or permeate. A leak may have both mechanisms operating in parallel.

5.1.14 *outboard leak rate* — leakage rate expressed in $\text{Pa}\cdot\text{m}^3/\text{s}$ ($\text{atm}\cdot\text{cc}/\text{sec.}$) occurring from inside to outside when an internal pressure is greater than the external pressure acting on the component or system. Outboard leakage is typically determined by introducing a tracer gas into the interior of the piping system or component under test.

5.1.15 *retainer* — a holding mechanism to keep a seal in place.

5.1.16 *seal* — a device (i.e., gasket, O-ring, etc.) that joins two elements or systems so as to prevent leakage.

5.1.17 *sealing system* — a system that consists of two mating surfaces (e.g., component/ substrate), seal(s), fasteners (screws), and any necessary hardware (e.g., seal retainer).

5.1.18 *substrate* — the block consisting of machined passage(s) which define the flow path of a gas. Gas control components are attached to certain areas on the substrate block with gas seals at the interface.

5.1.19 *surface-mounted gas systems* — term used to denote the gas distribution technology where surface-mounted gas components (e.g., filters, regulators, mass flow controllers, and valves) are mounted onto a flat substrate which defines the flow path of the gas. The sealing system will commonly be located at various locations within the interface plane between component and substrate.

5.1.20 *test pressure* — the pressure at which a sealing system is hydrostatically tested. The test pressure is commonly defined as $1.5\times$ the maximum design pressure.

5.1.21 *vibration table* — a mechanized table that will vibrate with a controlled frequency, direction(s), and amplitude. It is commonly used for vibration testing.

6 Significance and Use

6.1 The following tests shall be performed on seals which have been assembled into a test fixture similar to the fixtures described in Related Information 1. These fixtures are not intended to duplicate an entire gas system, but are established to evaluate seals only.

6.2 All sealing surfaces and fastening systems shall be manufactured in strict accordance with seal manufacturer's instructions and requirements. In addition, seals shall be handled and installed per seal manufacturer's instructions.

7 Universal Test Methods

7.1 The tests listed in this section shall be performed as a minimum for all sealing systems. This section defines tests that pertain to both surface-mounted and conventional gas sealing systems.

NOTE 1: Refer to the Related Information section for conventional and surface-mount test fixtures.

7.2 Sample configuration shall be defined prior to testing for each section and noted with test results.

7.3 Helium Leak Tests, Operating and Proof Pressure

7.3.1 Inboard Helium Leak Test

7.3.1.1 Testing shall be conducted per test methods and procedures outlined in SEMI F1 test methods at room temperature.

7.3.1.2 Sample quantity: 20

7.3.2 Outboard Helium Leak Test and Proof Test

7.3.2.1 Testing shall be conducted per procedures outlined in SEMI F1 method 2 and the following paragraphs.

7.3.2.2 Assemble the sample per manufacturer's instructions.

7.3.2.3 Connect the leak detector to the vacuum chamber and evacuate the chamber.

NOTE 2: The chamber must be equipped with over pressure protection in the event of failure.

7.3.2.4 Gradually increase pressure of the test assembly to manufacturer's maximum rated working pressure and hold for two minutes. If leakage beyond acceptance requirements occurs at any time during testing, note the pressure and leak rate and discontinue testing of that sample.

7.3.2.5 Continue increasing pressure to $1.5\times$ the manufacturer's maximum rated working pressure and hold for five minutes. Reduce the pressure back to the maximum rated working pressure and note the leak rate at this pressure.

7.3.2.6 Sample quantity: 4

NOTE 3: Outboard leak testing should be completed on only the referenced four samples. All subsequent leak testing required in conjunction with other testing should be inboard only.

7.4 Hydraulic Burst Test

7.4.1 Assemble the test sample per manufacturer's instructions.

7.4.2 Attach the sample to a hydraulic pressure source using qualified high pressure connections.

7.4.3 Samples shall be tested within an enclosure suitably designed to protect personnel from failure.

7.4.4 Test medium shall be water or hydraulic fluid.

7.4.5 Gradually increase pressure to maximum rated working pressure and hold for one minute.

7.4.6 Gradually increase pressure until failure, or $4\times$ manufacturer's rated working pressure is reached.

7.4.7 Sample quantity: 4

7.5 Temperature Cycle Test Method

7.5.1 Perform an initial inboard leak test on the sealing system in accordance with § 7.3.1.

7.5.2 Install the plumbing and test sample apparatus with test sample(s) installed into a temperature controlled chamber. Attach a thermocouple to the exterior of the apparatus adjacent to the test sample.

7.5.3 Heat the chamber until the temperature indicated by the thermocouple is $100 + 10/-0^{\circ}\text{C}$. Hold 10 minutes after the temperature stabilizes, then perform an inboard leak test in accordance with § 7.3.1.

7.5.4 Cool the chamber until the temperature indicated by the thermocouple is $-10 + 0/-10^{\circ}\text{C}$ and perform an inboard leak in accordance with § 7.3.1 while at temperature.

NOTE 4: If the seal is intended for outdoor service, refrigerate the test apparatus to $-54 + 0/-10^{\circ}\text{C}$.

7.5.5 Repeat ¶¶ 7.5.1 through 7.5.4 for a total of 5 cycles.

7.5.6 Sample quantity: 3 samples

7.6 Repeatability of Sealing System

7.6.1 Photograph mating surfaces at $10\times$ magnification.

7.6.2 Perform initial inboard leak test on sealing system per § 7.3.1.

7.6.3 A sequence of 10 make and remake cycles shall be performed on the same sealing system.

NOTE 5: Replace seal after each cycle if recommended by manufacturer.

7.6.4 Perform internal leak test per § 7.3.1 after each make and remake cycle.

7.6.5 Record all visible damage of the mating surfaces in the form of scratches, burrs, or other particles photographed after the different intervals at $10\times$ magnification.

7.6.6 Repeat ¶¶ 7.6.2 through 7.6.4 three additional times using a new set of mating surfaces each time.

NOTE 6: Replace seal after each cycle if recommended by manufacturer.

7.7 Shock and Vibration Test Method

7.7.1 This test is designed to evaluate the effects of random accelerations and sustained vibrations during shipment of gas systems.

7.7.2 Install seals and assemble per manufacturer's instructions. Refer to Related Information 1, Figure R1-1 through Figure R1-7 for test fixtures.

7.7.3 Perform inboard leak test per § 7.3.1.

7.7.4 Conduct shock test per MIL-STD-810E, Method 516.4, § I-3.1a, procedure I (functional shock), using terminal-peak saw-tooth shock pulse for ground equipment operation test.

7.7.5 Perform inboard leak test per § 7.3.1.

7.7.6 Conduct vibration test per MIL-STD-810E, Method 514.4, procedure 1 (basic transportation). Test duration: 1 hour per 1,609 km (1000 miles) of transportation in each directional axis.

7.7.7 Perform inboard leak test per § 7.3.1.

7.7.8 Sample quantity: 3

7.8 Seal Preload Safety Factor

7.8.1 This test evaluates the effects of under-tightening and over-tightening a seal connection.

7.8.2 Install seal and assemble to 80% of the manufacturer's sealing load (e.g., torque).

7.8.3 Perform inboard leak test per § 7.3.1.

7.8.4 Disassemble and repeat using a new seal for each test.

7.8.5 Sample quantity: 3

7.8.6 Using the same components, install a new seal and assemble to 120% of the manufacturer's sealing load.

7.8.7 Perform inboard leak test per § 7.3.1, and note any form of deformation or damage to the seal connection. Replace connection for subsequent tests if connection is damaged.

7.8.8 Disassemble and repeat using a new seal for each test.

7.8.9 Sample quantity: 3

7.9 Torsion Test

7.9.1 This test is designed to measure whether the sealing system can maintain leak integrity when torque is applied to an adjacent component or position.

7.9.2 Install seal and assemble per manufacturer's instructions. Refer to Related Information 1, Figure R1-1, through Figure R1-7 for test fixtures.

7.9.3 Place a stationary digital torque wrench on the adjacent component position to measure the torque applied as make up takes place. Location of placement is seen in Figure R1-1.

7.9.4 Test per § 7.3.1.

7.9.5 Rotate the adjacent component in a clockwise direction until 57.6 kgf-cm (50 lbf-in; 5.649 N·m) torque is measured on the stationary torque wrench.

7.9.6 Test per § 7.3.1.

7.9.7 Rotate the adjacent component in a counterclockwise direction until 57.6 kgf-cm (50 lbf-in; 5.649 N·m) torque is measured on the stationary torque wrench.

7.9.8 Test per § 7.3.1.

7.9.9 Repeat ¶¶ 7.9.5 through 7.9.8 with 115.2 kgf-cm (100 lbf-in; 66.355 N·m) torque.

7.9.10 Sample quantity: 3

7.10 *Surface Defect Test*

7.10.1 This Test Method is for evaluating the robustness of sealing technologies for gas delivery systems used in semiconductor manufacturing against surface defects which could occur during normal handling in the field.

7.10.2 A material testing machine is used to make a defect on the surface. In place of a pyramid-shaped indenter for hardness measurement, a wedged shaped indenter, specifically designed for this test, is installed in the adjustable-load hardness measurement equipment shown in Figure 1 and is used to make a surface defect on test sample.

7.10.3 The indenter shall meet the dimensional requirement shown in Figure 2. The indenter shall have a minimum hardness of 500 Vickers.

7.10.4 This test defaces only the seal face of the component, and determines the size of the surface defect as well as the corresponding leak. In this way, the degree of weakness of the component's seal face, as well as the extent to which the leak can be stopped depending on defect size, can be determined, making it possible to evaluate the robustness of the sealing technology being tested.

7.10.5 Equipment shown in Figure 1 and Figure 2 is used to make a defect on the surface of the seal face of the component or substrate.

7.10.6 Load levels are 2, 4, 6, 8 kgf, (19.61, 39.23, 58.84, 78.45 N) and add 2 kgf (19.61 N) incrementally until leaking occurs. The test sample should be tightened according to the manufacturer's installation manual.

7.10.7 A leak test is performed on the sample according to the procedure in SEMI F1. As this document is a test method, not a specification, only the procedure in SEMI F1 applies.

7.10.8 Correlation between the load and the leak is determined.

7.10.9 Surface defect size (depth and width) is measured using surface roughness measuring equipment for reference.

7.11 *Particle Cleanliness After Seal Make-up*

7.11.1 The purpose of this test is to verify the particles generated during a sealing-system compression cycle. This test involves testing for particles in situ, while compressing a sealing system.

7.11.2 Test per SEMASPEC 90120390B-STD. Use a flow-through testing apparatus that is seal-specifically designed for use in this test, in place of a valve.

7.11.3 Testing must be performed in a Class 100 (or better) environment in order to obtain low background counts before the seals are tightened to the substrate.

7.11.4 Follow test protocol in SEMASPEC 90120390B-STD with the exception that a flow-through bypass fixture must be attached 'finger-tight' to a test substrate before obtaining a background count.

7.11.5 The testing apparatus with the seals to be tested must be purged with 0.01 μm filtered gas for sufficient time so as to remove all inherent particles present during installation.

7.11.6 Once the lowest possible background count has been generated, the flow-through test device must be tightened to the seal manufacturer's specifications while gathering particle counts from the particle counter. It is recommended that the counts are recorded via a data acquisition device to allow the user to compress the seals while the particles are being recorded.

7.11.7 This test is for comparison purposes only between data generated at a common facility with common test setup.