

K $\beta$  peak with Fe K $\alpha$  peak interferes with determination of low levels of Manganese in Iron-Chromium matrix.

## 8 Apparatus

8.1 *Instrumentation* — Any SEM instrument used for this method must be capable of a minimum point-to-point resolution of 30 nm, as measured with a NIST<sup>4</sup> traceable standard. Any commercially available SEM with image recording capabilities of at least 100 cm<sup>2</sup> image and an EDS analyzer capable of 170 eV or less FWHM resolution for Mn K $\alpha$  may be employed.

8.2 The SEM shall have a sample stage capable of aligning the sample to provide multiple areas of view with the orientation of the electron beam approximately normal to the surface, and to optimize secondary electron and X-ray signal collection efficiencies.

8.3 *Grid Overlay* — A transparent grid overlay of grid size equal to 1.814 micrometers multiplied by the pre-selected magnification in the range 3000 to 3600 (grid size would be 6.35 mm [= ¼ inch] square for 3500 $\times$  magnification). The grid lines shall be as fine as possible but clearly visible. The grid overlay shall be designed to overlay the image of a photomicrograph with 252 grids (18 by 14 grids for the 3.5 by 4.5 inch image of a standard Polaroid photomicrograph) with minimal interference from alphanumeric information recorded with the photomicrograph, but without reference to any features in the photomicrograph. The rows and columns of the grid overlay may be indexed with alphanumerics to identify and locate specific features in the photomicrographs (see examples in Appendices 1 and 2). It is suggested that a fixed reference point for the edges of the photomicrograph be established to be consistently used in all overlays.

## 9 Reagents and Materials

9.1 *Sample Preparation Materials* — Equipment required to section the test specimen without damaging or contaminating the surfaces to be analyzed. A clean, dry hacksaw or dry bandsaw is recommended, using a slow cutting speed to avoid excessive sample heating.

9.2 *Sample Mounting Materials* — Sample mounting holders specific to the SEM instrument used. Conductive paste or tape used to adhere the sample to the holder must be vacuum stable and applied so that the area of analytical interest is not contaminated.

## 10 Safety Precautions

10.1 This test method does not purport to address the safety considerations associated with use of high voltage, vacuum, electron and X-ray producing equipment.

10.2 The method assumes a SEM and EDS analyst with adequate skill level as well as knowledge of instrumentation and associated safety precautions.

## 11 Test Specimen

11.1 Specimens are to be sectioned to appropriate size for the particular SEM instrument. Any sample preparation technique used shall avoid introducing contamination onto the surface to be measured. In addition, preparation must avoid heating of the sample in excess of approximately 100°C to avoid oxide growth or change in surface composition. Slow speed dry cutting is recommended.

11.2 After preparation, samples shall be protected from contamination by means such as sealing in noncontaminating bags or wrapping in clean aluminum foil. Appropriate cleaning of the analysis surface to remove sample preparation residues is allowed but not required. Any such cleaning procedures should be stated in the report. After sectioning, samples should be analyzed promptly.

11.3 The samples shall be analyzed in the uncoated condition; ie, without sputtering with a conductive coating.

## 12 Preparation of Apparatus

12.1 The SEM shall be in good condition to assure proper performance in accordance with the manufacturer's specifications.

## 13 Calibration and Standardization

13.1 Instrument calibration for magnification and EDS performance shall be performed in accordance with ASTM E 766 and ANSI-IEEE 759 and/or the instrument manufacturer's recommendations. The magnification should be calibrated at the working distance selected. Calibration frequency shall be per instrument manufacturer's recommendations.

## 14 Procedure

14.1 The sample is to be mounted in accordance with manufacturer's recommendations and in a manner consistent with high vacuum analytical procedures. The sample shall be oriented such that the areas of interest for SEM imaging can be viewed at 70 to 90° incidence angle of the electron beam to the surface with optimum secondary electron collection efficiency, and the ability to re-orient the sample for optimization of X-ray

<sup>4</sup> National Institute of Standards and Technology, 100 Bureau Drive, Stop 3460, Gaithersburg, MD 20899, (301) 975-NIST (6478) TTY (301) 975-8295, website: www.nist.gov.

collection efficiency. Any adhesives used to mount samples must not contaminate the surface to be analyzed. To remove loosely adhered surface particles, the sample may be blown off immediately before introduction into the SEM with clean, dry, 0.1 micrometer filtered gas.

14.2 Place the sample in the SEM chamber for pumpdown. Activate the electron beam when vacuum conditions meet manufacturer's recommendations. The instrument accelerating voltage should be a consistently selected value within the range 15 to 25 KeV; 20 KeV is suggested. The working distance should be within the range recommended by the instrument manufacturer. Sample position (tilt angle and orientation) may be adjusted to optimize EDS detector collection efficiency for EDS analysis.

14.3 Increase the magnification to  $\geq 400\times$  for initial focus; adjust instrument parameters for astigmatism and other anomalies. Decrease magnification to  $200\times$  and move the sample while viewing until an area judged as representative of the whole is in view. The area to be analyzed should be as free of particles and defect features produced by sample preparation as possible.

14.4 Refocus and record images of this area at  $200\times$ ,  $1000\times$ , and a pre-selected, consistently used magnification within the range 3000 to  $3600\times$ . Move to another representative area and record an image at the same 3000–3600 $\times$  magnification. Move to a third representative area and record another image at the 3000–3600 $\times$  magnification.

14.5 Each area selected for recording should include a defect or metallurgical feature to demonstrate proper focusing, contrast adjustment and resolution capability. Contrast adjustment should be performed per the technique described in Section 4.7.2.1 of *Scanning Electron Microscopy and X-Ray Microanalysis*.<sup>5</sup> All defects and/or features for which the change in signal  $\Delta S$  due to the contrast exceeds the noise  $N$  by a factor of 5 minimum must be clearly distinguishable in the image recorded (see Section 4.5 of reference 5).

14.6 If any inclusions or contamination are noted in the 3000–3600 $\times$  images, EDS analysis of representative defect(s) may be performed if requested by the company for which the test method is performed. Collect X-ray signals for a sufficient length of time to obtain an integrated count of  $\geq 250,000$  within the range of 0 to 10 keV.

---

<sup>5</sup> Scanning Electron Microscopy and X-Ray Microanalysis: A Text for Biologists, Materials Scientists, and Geologists; Joseph I. Goldstein, Dale E. Newbury, Patrick Echlin, David C. Joy, A. D. Romig, Jr., Charles E. Lyman, Charles Fiori, Eric Lifshin; Second Edition, Plenum Press, New York (1992).

## 15 Calculations or Interpretation of Results

15.1 The 3000–3600 $\times$  recorded images shall be placed under the transparent overlay grid. The number of grid squares that contain all or part of a surface defect shall be counted and summed for each photomicrograph. Each such grid square shall only be counted once; it is not the intent of this Test Method to count individual defects. If a defect appears in more than one adjacent grid square, each grid square occupied shall be counted.

NOTE 2: Examples of scanning electron micrographs overlaid with an alphanumeric indexed grid showing specific types of defects with comments on their locations and whether they should be counted may be viewed at <http://www.semi.org/web/wstandards.nsf/url/SurfaceAnalysisExamples>.

15.2 Surface film residues resulting from improper cleaning are generally diffuse and difficult to quantify. These residues, if present, are counted as one per image. Particles that loosely adhere to the surface must be presumed to be artifacts of sample preparation or exposure, and therefore shall be ignored.

15.3 Peaks appearing in the EDS spectra shall be identified and the spectra labeled to indicate whether it is a representative area, or identified as a defect from a photomicrograph.

## 16 Reporting Results

16.1 A tabular summary of defects counted per 3000–3600 $\times$  photomicrograph shall be presented. Additionally, the table should indicate an average of defect counts and the maximum from the three 3000–3600 $\times$  images. An example of a table reporting defect counting results and the photomicrographs from which the defect counts were taken are shown in Appendix 1.

16.2 All photomicrographs are to be permanently labeled with sample identification, magnification, a magnification scale bar, date, and analyst identification. The photomicrographs should be available for inspection upon request.

16.3 EDS spectra are to be labeled with the same information, plus peak identifications. If any instrument parameters (e.g., tilt angle) were changed for EDS analysis, these must be noted. If peak height differences are such that adequate representation of all peaks cannot be made from the same plot, two plots having different scaling shall be included. Identification of each defect analyzed, either by arrow on photomicrograph or by notation of grid position, shall be made.



## **17 Related Documents**

### *17.1 ASTM Standards*

ASTM F1372-93 — Standard Test Method for Scanning Electron Microscope (SEM) Analysis of Metallic Surface Condition for Gas Distribution System Components

ASTM F1375-92 — Standard Test Method for Energy Dispersive X-Ray Spectrometer (EDX) Analysis of Metallic Surface Condition for Gas Distribution System Components

## APPENDIX 1

### EXAMPLE OF AN ACCEPTABLE FORMAT FOR REPORTING DEFECT COUNTING RESULTS

NOTE: The material in this appendix is an official part of SEMI F73 and was approved by full letter ballot procedures.

**Table A1-1 Example of an Acceptable Format for Reporting Defect Counting Results**

Analyst Name: John Doe

Analytical Service Provider: ABC Services Lab

Date: May 10, 1999

(Customer; Order Number; Source): Pure Products, Inc.

<i>Sample</i>	<i>Micro No.</i>	<i>Defect Count</i>	<i>Comments (Location indexing optional)</i>	<i>Average Count</i>	<i>Maximum Count</i>
A22-6/5/97	1 (from Figure A1-1)	13	Inclusion J7, K7, I8, J8, K8; Pits A8, C7 and C8, E9, F8, J9, I12, I15	16	30
	2 (from Figure A1-2)	30	Stringer G14 to P3; Pit C13 and C14		
	3 (from Figure A1-3)	4	Pits D10 and E10, G13. Grain, twin boundaries, white sample prep debris not counted.		
(Sample #2 Identification)	1	12	Pits; stringer	17	23
	2	23	Scratch		
	3	16	Inclusion		
(Sample #3 Identification)	1	9	Stringer	6	9
	2	4			
	3	6	Pits		



Figure A1-1

Sample A22-6/5/97 – Micro 1 - Inclusion J7, K7, I8, J8, K8; Pits A8, C7 and C8, E9, F8, J9, I12, I15.

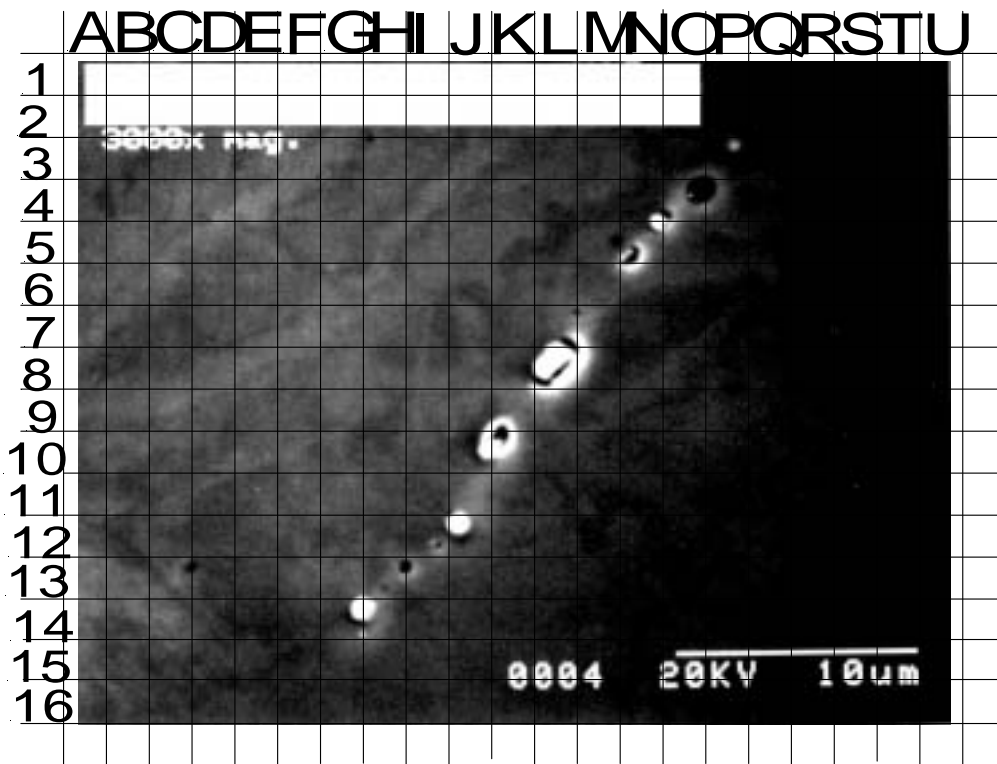
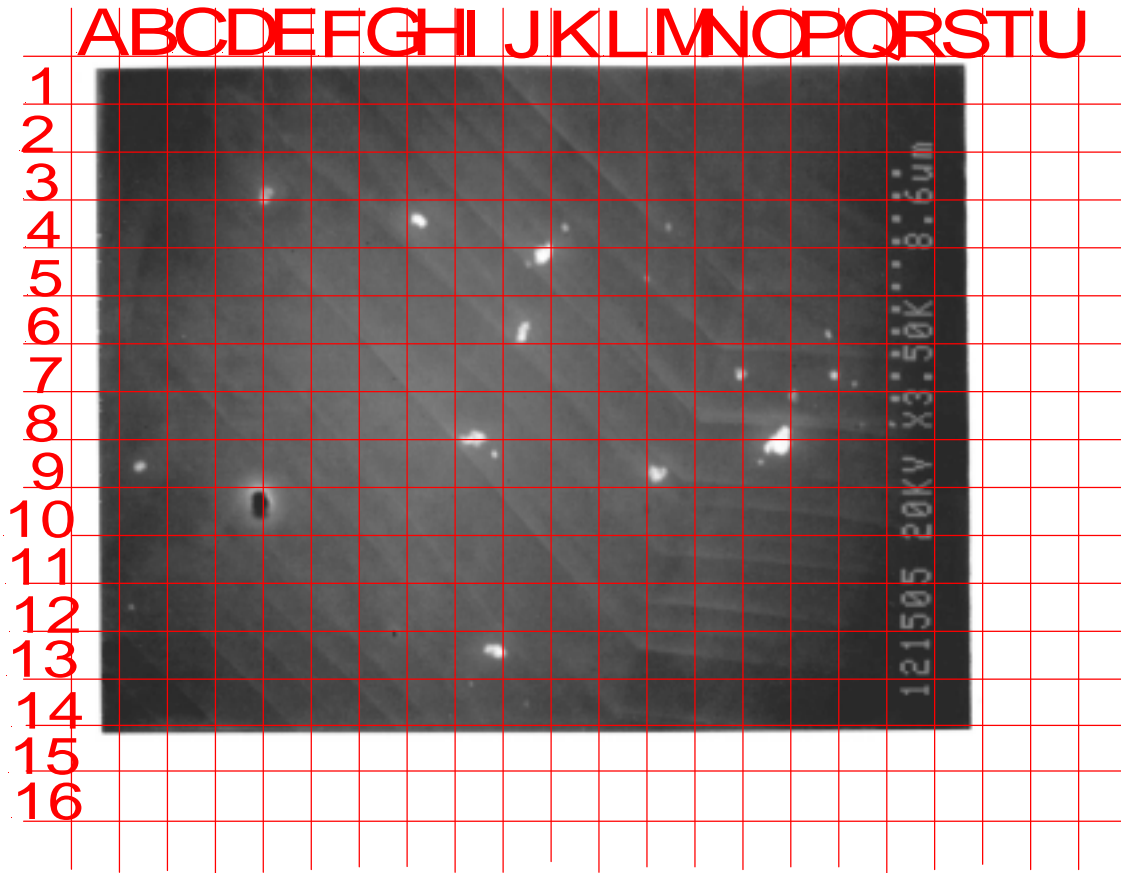


Figure A1-2

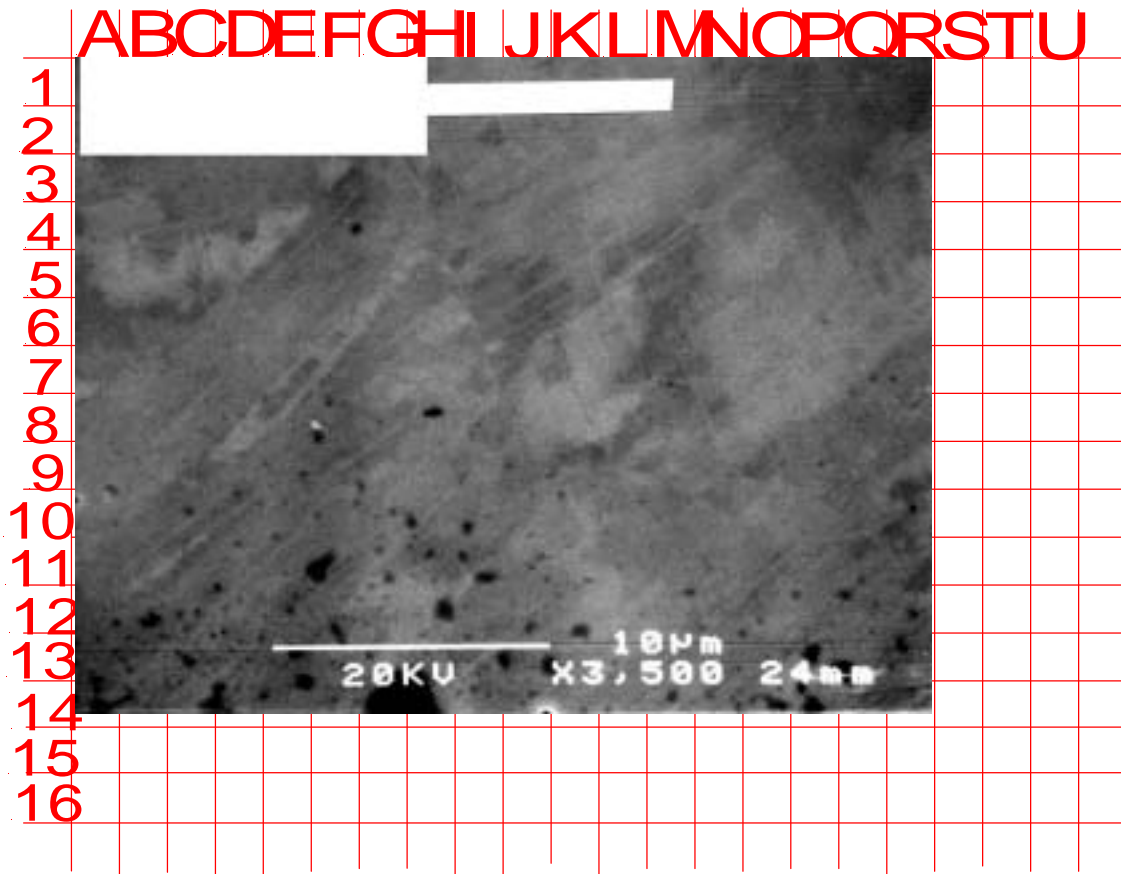
Sample A22-6/5/97 – Micro 2 - Stringer G14 to P3; Pit C13 and C14



**Figure A1-3**  
**Sample A22-6/5/97 – Micro 3 - Pits D10 and E10, G13. Grain, Twin Boundaries, White Sample Preparation**  
**Debris Not Counted.**

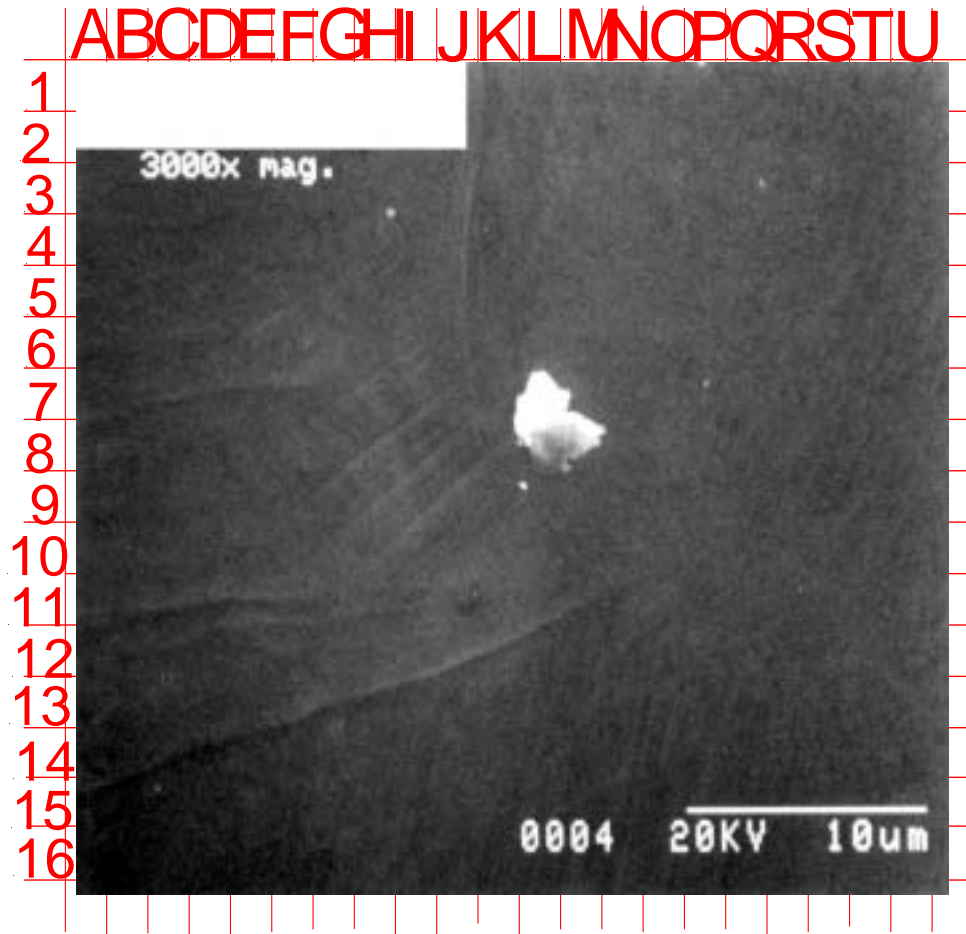
## APPENDIX 2 EXAMPLE OF DEFECTS

NOTE: The material in this appendix is an official part of SEMI F73 and was approved by full letter ballot procedures.



**Figure A2-1**

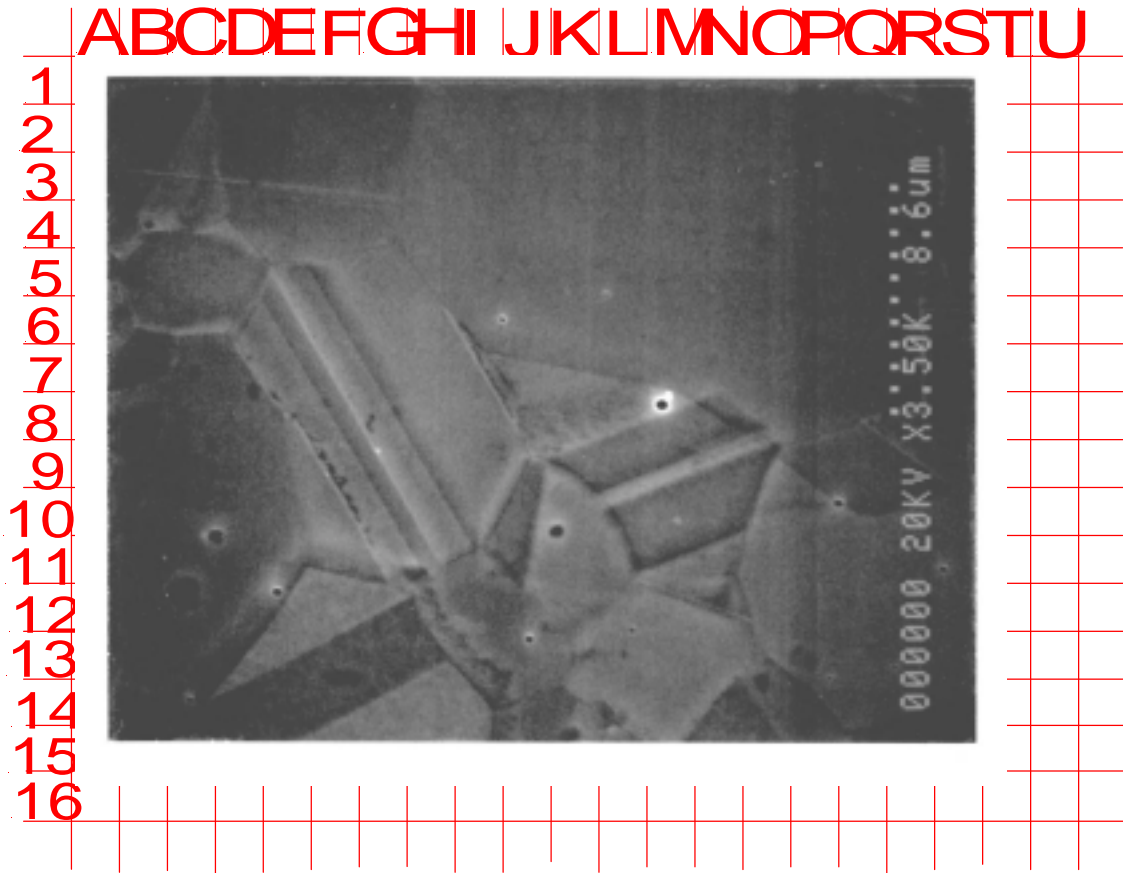
**Diffuse Black Spots in Grids F4, H8, and Along Rows 11 Through 14 are Believed to be Surface Contamination, Probably Hydrocarbon, and Should be Counted. This Type of Contamination Should be Apparent in Surface Analysis (Auger or ESCA) if it is Not an Artifact of Sample Preparation. The “Mottled” Appearance Should Not be Counted.**



**Figure A2-2**

**Large Particle in Grids K, L, M, 7 and 8 is Assumed to be an Example of Debris from Sample Preparation and Should Not be Counted. Lightly Etched Grain and Twin Boundaries in Grids G Through J, 6 Through 13 are Not Significant Defects and are Not Counted.**





**Figure A2-3**  
**Heavily Etched Grain and Twin Boundaries; Worst Cases Should be Counted – E5 Through H15; I and J 9, 10 and 11; K9 Through P10. Also Pits at P3, L5, I6, M8, P10, S11, J10 and K10, C10, C11, D10, D11, E12, J13, L12, C14.**

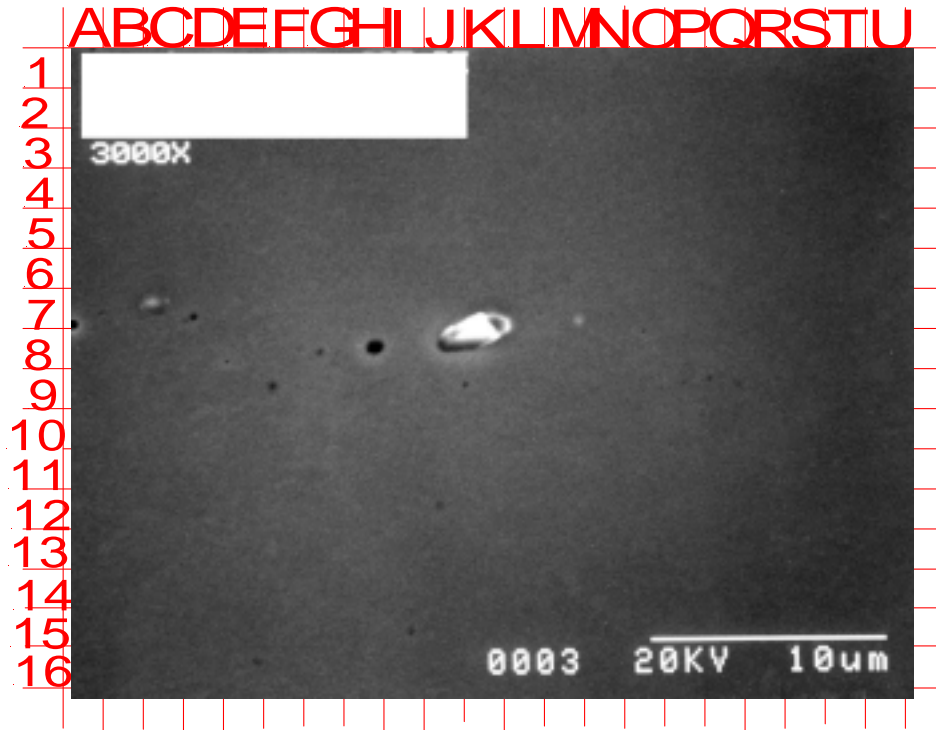
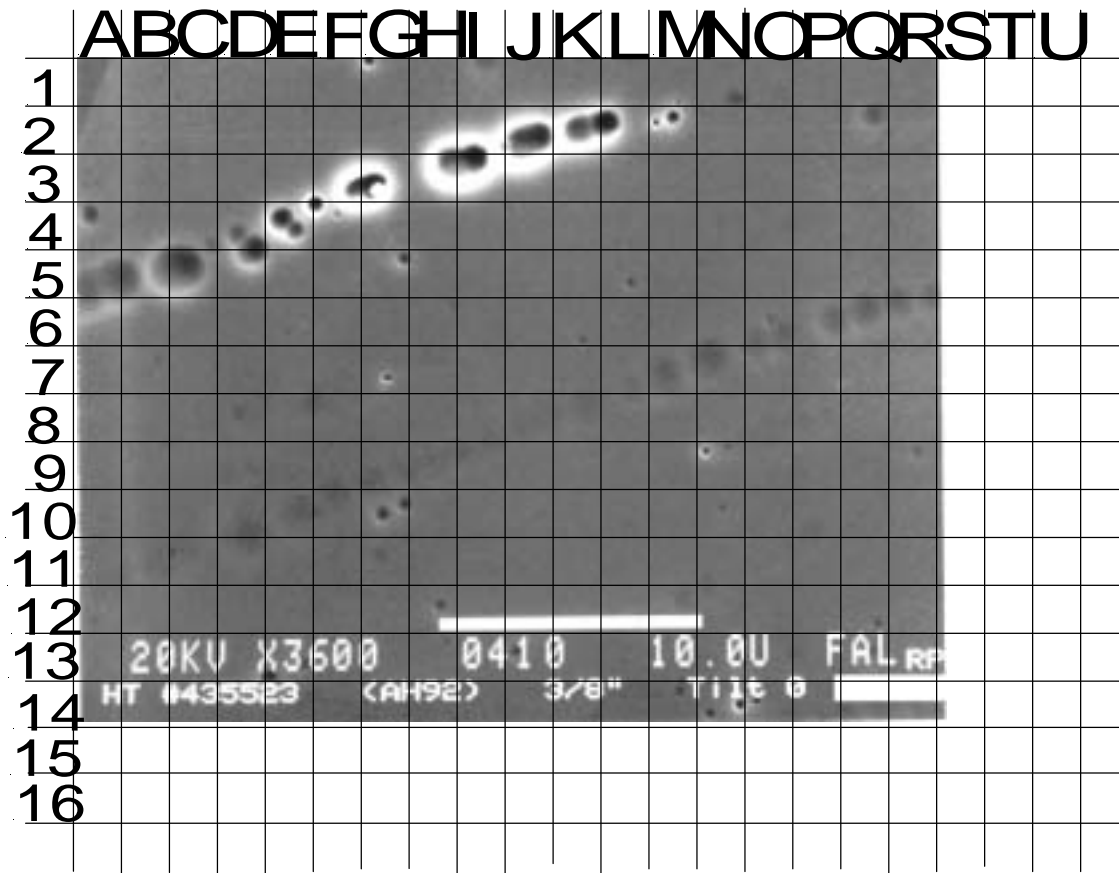


Figure A2-4

Large Inclusion is Counted in Grids J7, K7, I8, J8 and K8. Pits in A8, C7 and C8 (Same Pit on Grid Boundary), E9, F8, J9, I12, I15.



**Figure A2-5**

**Pits Left by Inclusion Stringers in Which the Inclusions Have Been Electropolished Out; Also Numerous Discrete Pits. All Pits with Edges Distinct Enough to Image with a “White Halo” Effect are Countable. This Includes All of the Pits Associated with the Upper Stringer, and Those in the Lower Stringer in Grids M7N7, O6 and O7, P6, Q6, R5 and R6. Also G1, Q1, A4, G5, L5, G7, N9, R9, G10, H12, Q12, E13, J13, N14, O14.**

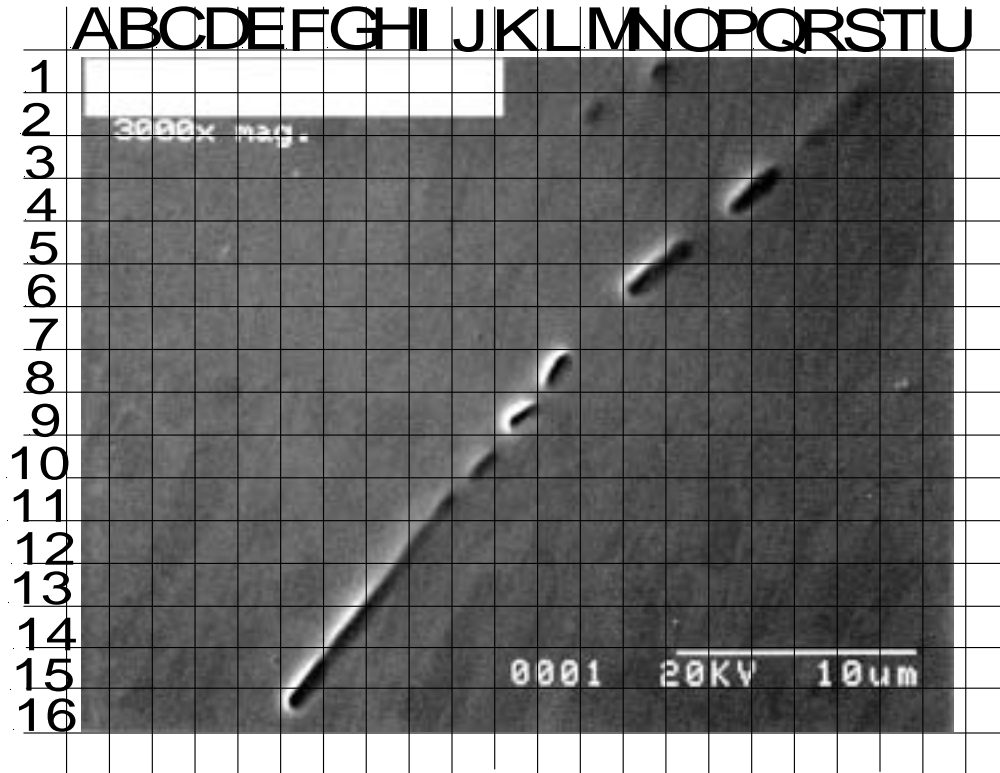
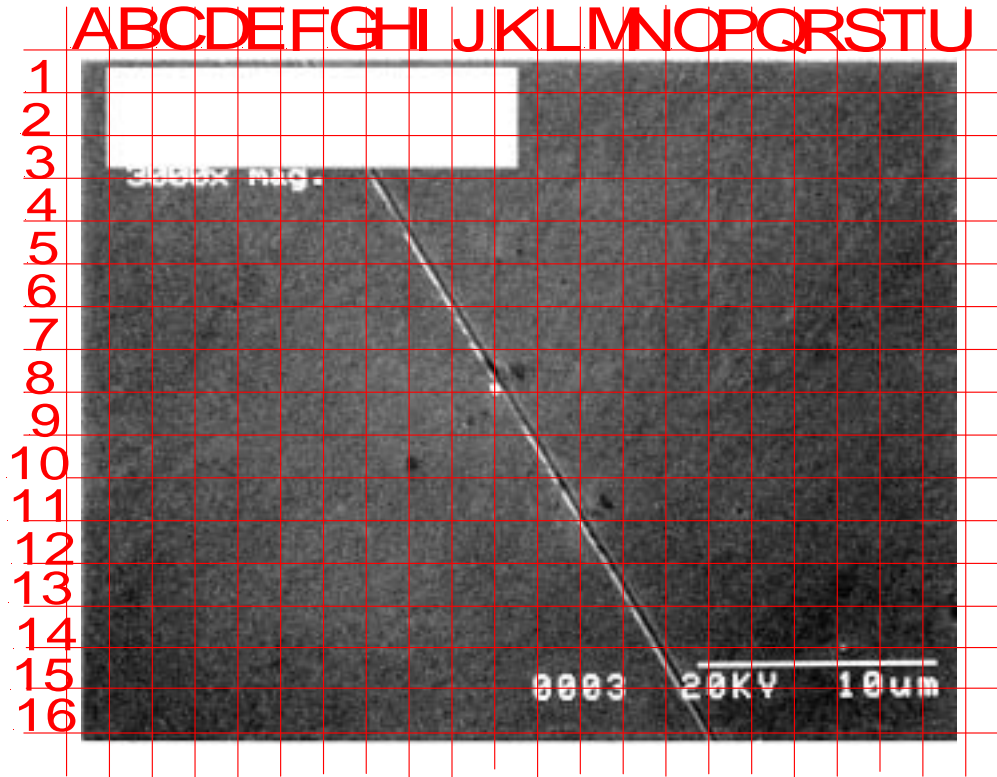


Figure A2-6

Long, Partially Continuous Stringer; the Inclusion that Formed this Stringer has been Electropolished Out.  
All Grids Occupied by the Stringer Should be Counted, from F16 to S2. Also Pits at M2, N1 and O1.



**Figure A2-7**  
**Scratch Counted in Grids H3-5, I5 and 6, J7 and 8, K8-10, L10 and 11, M12 and 13, N13-15, O15 and 16.**  
**Pits Counted in Grids J9, H10, I10, M11.**

**NOTICE:** SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer' s instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

By publication of this standard, Semiconductor Equipment and Materials International (SEMI) takes no position respecting the validity of any patent rights or copyrights asserted in connection with any items mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights are entirely their own responsibility.

# SEMI F74-1103

## TEST METHOD FOR THE PERFORMANCE AND EVALUATION OF METAL SEAL DESIGNS FOR USE IN GAS DELIVERY 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 September 16, 2003. Initially available at [www.semi.org](http://www.semi.org) October 2003; to be published November 2003. Originally published November 2002.

### 1 Purpose

1.1 This document 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:** 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 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 document 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

#### 4.1 SEMI Standards

SEMI E49 — Guide for Standard Performance, Practices, and Sub-Assembly for High Purity Piping Systems and Final Assembly for Semiconductor Manufacturing Equipment

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

#### 4.2 ASME Standards<sup>1</sup>

B31.3 — ASME Code for Process Piping

#### 4.3 Military Standard<sup>2</sup>

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

#### 4.4 Federal Standard<sup>3</sup>

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

#### 4.5 ISO Standards<sup>4</sup>

ISO 14644-1 — Cleanrooms and associated controlled environments -- Part 1: Classification of air cleanliness

ISO 14644-2 — Cleanrooms and associated controlled environments -- Part 2: Specifications for testing and monitoring to prove continued compliance with ISO 14644-1

<sup>1</sup> American Society of Mechanical Engineers, Three Park Avenue, New York, NY 10016-5990, USA. Telephone: 800.843.2763 (U.S./Canada), 95.800.843.2763 (Mexico), 973.882.1167 (outside North America), Website: [www.asme.org](http://www.asme.org)

<sup>2</sup> Available through the Naval Publications and Forms Center, 5801 Tabor Avenue, Philadelphia, PA 19120-5099, USA. Telephone: 215.697.3321

<sup>3</sup> Federal Standard, c/o U.S. Government Printing Office, Washington DC 20402

<sup>4</sup> International Organization for Standardization, ISO Central Secretariat, 1, rue de Varembé, Case postale 56, CH-1211 Geneva 20, Switzerland. Telephone: 41.22.749.01.11; Fax: 41.22.733.34.30, Website: [www.iso.ch](http://www.iso.ch)

ISO 14644-4 — Cleanrooms and associated controlled environments -- Part 4: Design, construction and start-up

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

## 5 Terminology

### 5.1 Abbreviations and Acronyms

5.1.1 *atm* — atmosphere

5.1.2 *cc* — cubic centimeter

5.1.3 *ft-lbs* — foot-pounds (force)

5.1.4 *kPa* — kiloPascal

5.1.5 *MFC* — Mass Flow Controller

5.1.6 *mPa* — megaPascal

5.1.7 *N* — Newton

5.1.8 *Nm* — Newton meters

5.1.9 *Pa* — Pascal

5.1.10 *psig* — pounds per square inch (gauge pressure)

### 5.2 Definitions

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

5.2.2 *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 0.5µm or larger.

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

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

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

5.2.6 *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.

5.2.7 *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.

5.2.8 *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.

5.2.9 *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.

5.2.10 *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.

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

5.2.12 *inboard leak rate* — leakage rate expressed in  $\text{Pa}\cdot\text{m}^3/\text{s}$  (*atm cc/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.

5.2.13 *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.

5.2.14 *outboard leak rate* — leakage rate expressed in  $\text{Pa}\cdot\text{m}^3/\text{s}$  (*atm-cc/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.

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

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

5.2.17 *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).

5.2.18 *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.

5.2.19 *Surface-mounted Gas Systems* — term used to denote the gas distribution technology where *surface-mounted* gas components (e.g. filters, regulators,

MFC's, 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.

**5.2.20 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.

**5.2.21 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

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.

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

### 7.2 Helium Leak Tests, Operating and Proof Pressure

#### 7.2.1 Inboard Helium Leak Test

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

7.2.1.2 Sample quantity: 20

#### 7.2.2 Outboard Helium Leak Test and Proof Test

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

7.2.2.2 Assemble the sample per manufacturer's instructions.

7.2.2.3 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.

7.2.2.4 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 anytime during testing, note the pressure and leak rate and discontinue testing of that sample.

7.2.2.5 Continue increasing pressure to 1.5× 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.

7.2.2.6 Sample quantity: 4

NOTE 3: Outboard leak testing should be completed on only the referenced 4 samples. All subsequent leak testing required in conjunction with other testing should be inboard only.

### 7.3 Hydraulic Burst Test

7.3.1 Assemble the test sample per manufacturer's instructions.

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

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

7.3.4 Test medium shall be water or hydraulic fluid.

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

7.3.6 Gradually increase pressure until failure, or 4x manufacturer's rated working pressure is reached.

7.3.7 Sample quantity: 4

### 7.4 Temperature Cycle Test Method

7.4.1 Perform an initial inboard leak test on the sealing system in accordance with Section 7.2.1.

7.4.2 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.

7.4.3 Heat the chamber until the temperature indicated by the thermocouple is  $100 \pm 10/0^{\circ}\text{C}$ . Hold 10 minutes after the temperature stabilizes, then perform an inboard leak test in accordance with Section 7.2.1.

7.4.4 Cool the chamber until the temperature indicated by the thermocouple is  $-10 \pm 0/-10^{\circ}\text{C}$  and perform an inboard leak in accordance with Section 7.2.1 while at temperature.

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



7.4.5 Repeat Sections 7.4.1 through Section 7.4.4 for a total of 5 cycles.

7.4.6 Sample quantity: 3 samples

#### 7.5 *Repeatability of Sealing System*

7.5.1 Photograph mating surfaces at 10× magnification.

7.5.2 Perform initial inboard leak test on sealing system per Section 7.2.1.

7.5.3 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.

7.5.4 Perform internal leak test per Section 7.2.1 after each make and remake cycle.

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

7.5.6 Repeat Section 7.5.2 through Section 7.5.4 three additional times using a new set of mating surfaces each time.

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

#### 7.6 *Shock and Vibration Test Method*

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

7.6.2 Install seals and assemble per manufacturer's instructions. Refer to Figures R1-1, R1-2a~c, and R1-3a~c for test fixtures.

7.6.3 Perform inboard leak test per Section 7.2.1.

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

7.6.5 Perform inboard leak test per Section 7.2.1.

7.6.6 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.

7.6.7 Perform inboard leak test per Section 7.2.1.

7.6.8 Sample quantity: 3

#### 7.7 *Seal Preload Safety Factor*

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

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

7.7.3 Perform inboard leak test per Section 7.2.1.

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

7.7.5 Sample quantity: 3

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

7.7.7 Perform inboard leak test per Section 7.2.1. and note any form of deformation or damage to the seal connection. Replace connection for subsequent tests if connection is damaged.

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

7.7.9 Sample quantity: 3

#### 7.8 *Torsion Test*

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

7.8.2 Install seal and assemble per manufacturer's instructions. Refer to Figures R1-1, R1-2a~c, and R1-3a~c for test fixtures.

7.8.3 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.

7.8.4 Test per Section 7.2.1.

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

7.8.6 Test per Section 7.2.1.

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

7.8.8 Test per Section 7.2.1.

7.8.9 Repeat 7.8.5 through 7.8.8 with 115.2 cm-kgf (100 in-lbf; 66.355 Nm) torque.

7.8.10 Sample quantity: 3

#### 7.9 *Surface Defect Test*

7.9.1 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.

7.9.2 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.

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

7.9.4 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.

7.9.5 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.

7.9.6 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.

7.9.7 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.

7.9.8 Correlation between the load and the leak is determined.

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

#### 7.10 Particle Cleanliness After Seal Make-up

7.10.1 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.

7.10.2 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.

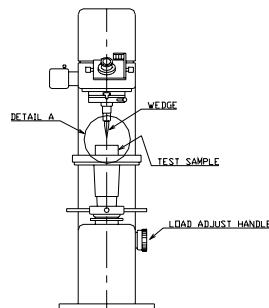
7.10.3 Testing must be performed in a Class 100 (or better) environment in order to obtain low background counts before the seals are tightened to the substrate.

7.10.4 Follow test protocol in Test Method for Particle Contribution (see Related Documents) with the exception that a flow-through bypass fixture must be attached "finger-tight" to a test substrate before obtaining a background count.

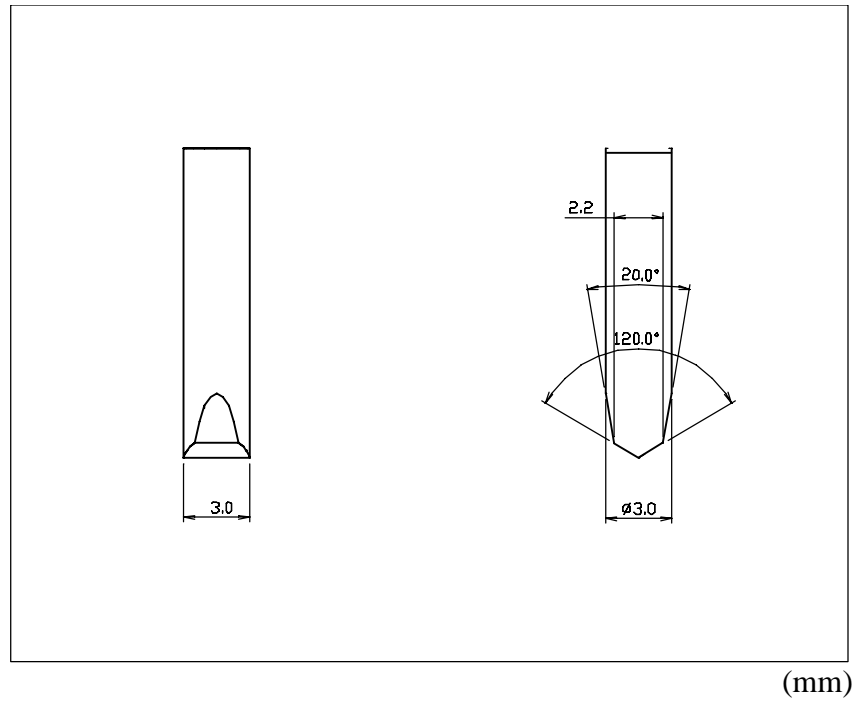
7.10.5 The testing apparatus with the seals to be tested must be purged with 0.01  $\mu\text{m}$  filtered gas for sufficient time so as to remove all inherent particles present during installation.

7.10.6 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.

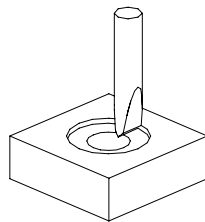
7.10.7 This test is for comparison purposes only between data generated at a common facility with common test setup.



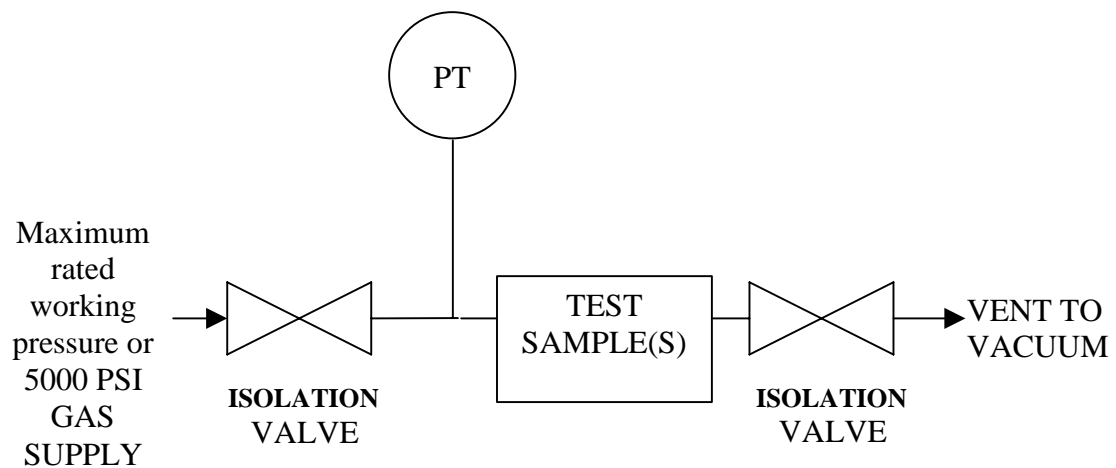
**Figure 1**  
**Load Adjustable Hardness Measuring Equipment**



**Figure 2**  
**Dimension of the Wedge-Shaped Indenter**



**Figure 3**  
**Wedge and Test Sample (Component or Substrate)**



**Figure 4**  
**Pressure Cycling Test Rig**

### 7.11 Corrosion Testing

7.11.1 Due to the lack of proven, standardized test methods for quantifying the performance of gas systems in corrosive environments, this document can only reference two applicable test procedures for use to supply qualitative information relating to the performance of gas system metal seals under corrosive serve: SEMI F77 and SEMASPEC 97043272A-TR (see Related Documents). It will be the responsibility of the requesting party to define the specific testing and acceptance criteria for this type of evaluation.

### 7.12 Pressure Cycling Test

7.12.1 Assemble the test sample(s) into the test rig shown in Figure 4 per the manufacturer's instructions.

7.12.2 Cycle the test sample(s) for a total of 250,000 cycles. The following represents one cycle:

7.12.2.1 Apply the manufacturer's maximum rated working pressure +/- 2% or 5000 +/- 100 psig, whichever is less.

7.12.2.2 Maintain pressure for at least 1 second after the transducer indicates that the pressure requirement has been achieved.

7.12.2.3 Vent the pressure to 74.5 KPa or less.

7.12.2.4 Maintain the vacuum for at least 1 second after the transducer indicates that vacuum requirement has been achieved.

7.12.3 Leak test per Section 7.2.1.

7.12.4 Sample quantity: 5

## 8 Related Documents

### 8.1 SEMATECH Documents<sup>5</sup>

SEMASPEC 90120390B-STD — Test Method for Particle Contribution

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

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

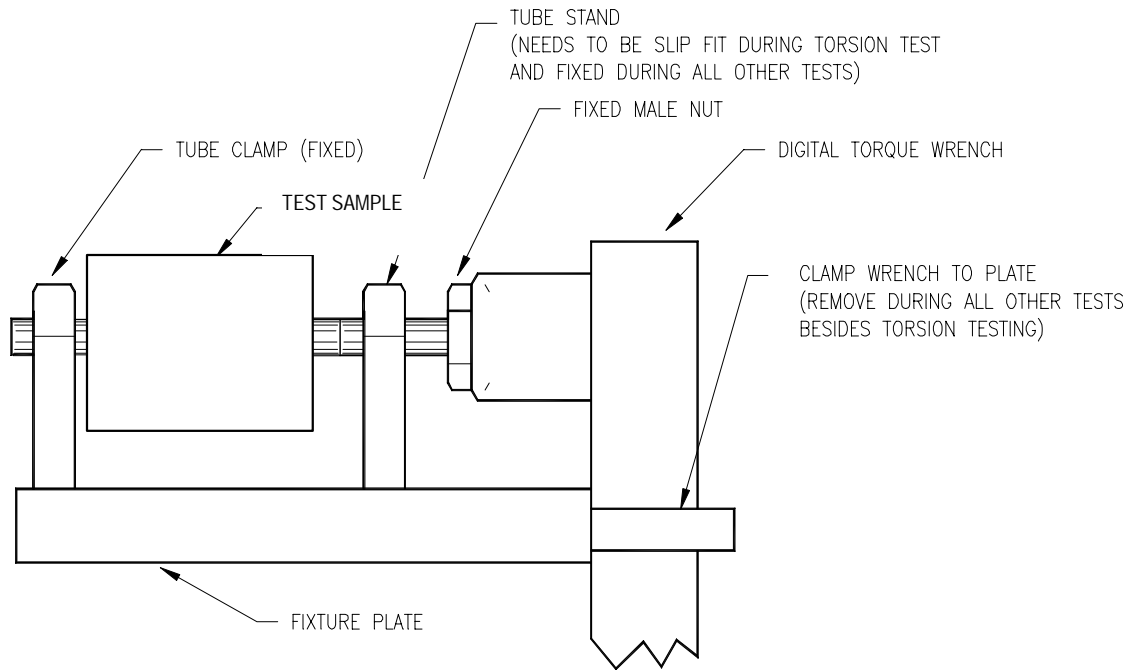
---

<sup>5</sup> SEMATECH, 2706 Montopolis Drive, Austin, TX, website: [www.sematech.org](http://www.sematech.org)

## RELATED INFORMATION 1

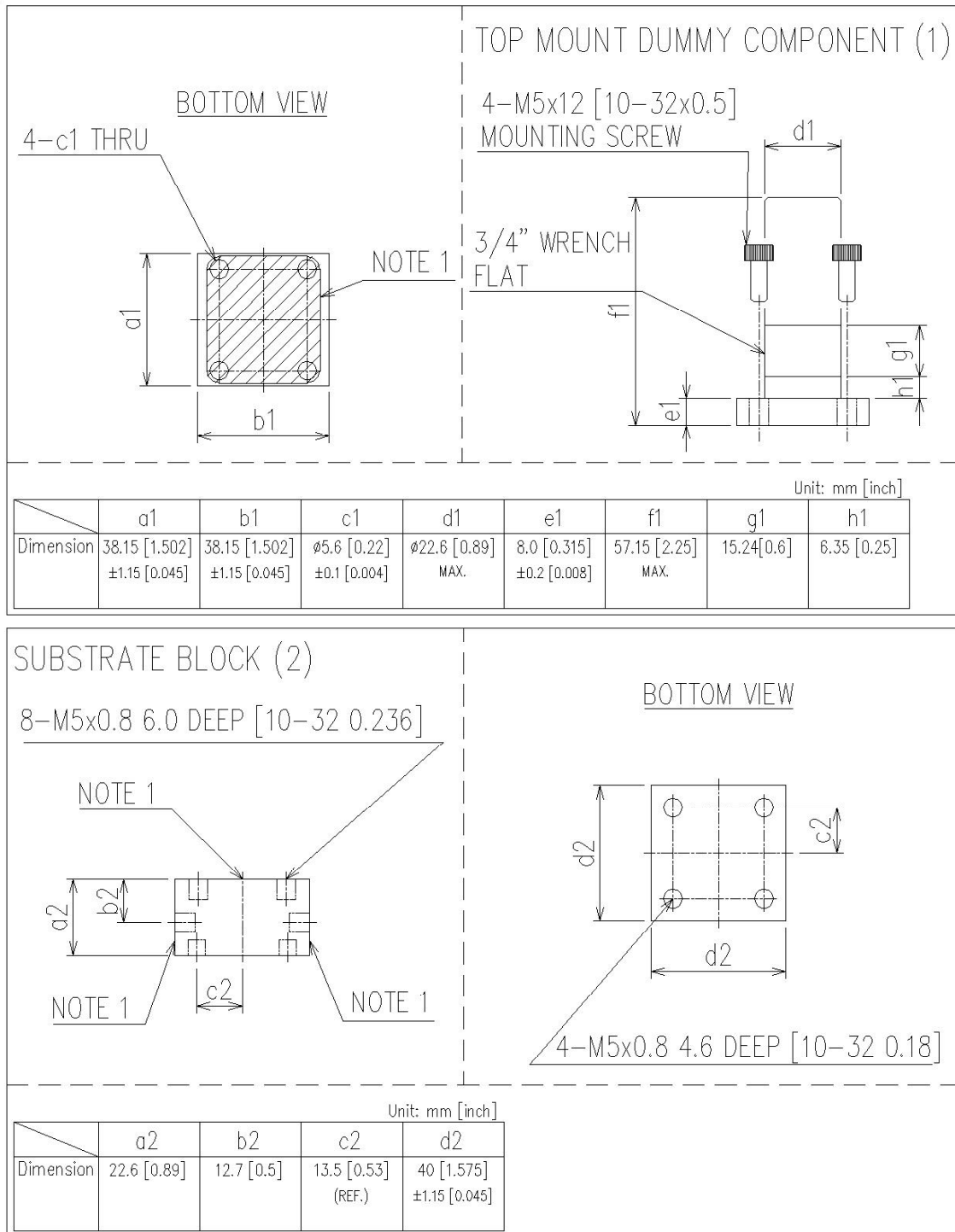
### SAMPLE PERFORMANCE TESTING FIXTURES

NOTE: This Related Information is not an official part of SEMI F74. The recommendation in this Related Information are optional and are not required to conform to this standard.



**Figure R1-1**  
**Conventional Performance Testing Sample**

# **COMPONENT VIEW; 1.5" VIBRATION/TORQUE TEST FIXTURE**

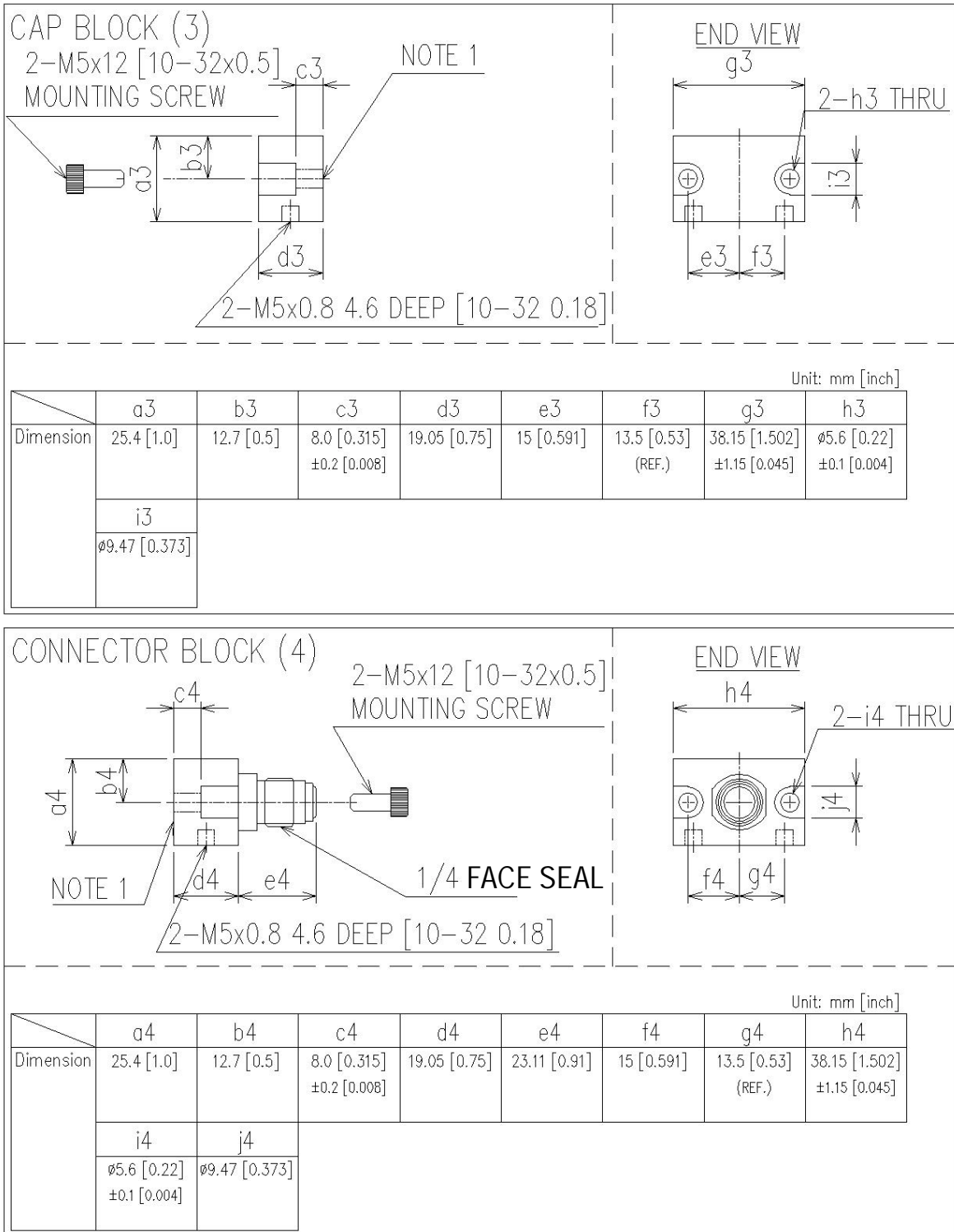


NOTE 1: Leak test port(s) and porting are dependent on seal system/substrate design configuration.

NOTE 2: Material of Construction: 316SS.

NOTE 3: Drawing scale is not specified.

**Figure R1-2a**  
**1.5" Surface-Mount Performance Testing Fixture**

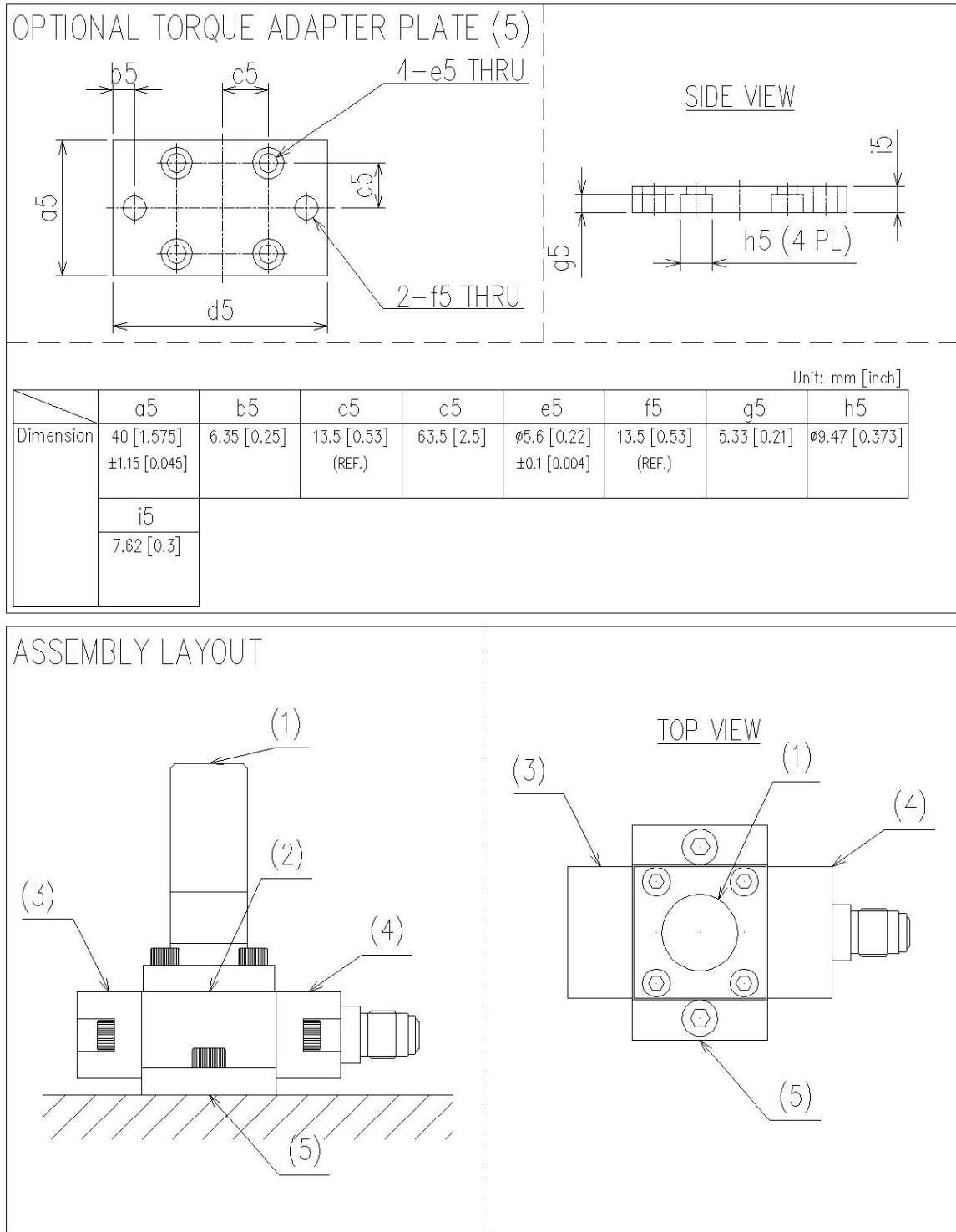


NOTE 1: Leak test port(s) and porting are dependent on seal system/substrate design configuration.

NOTE 2: Material of Construction: 316SS.

NOTE 3: Drawing scale is not specified.

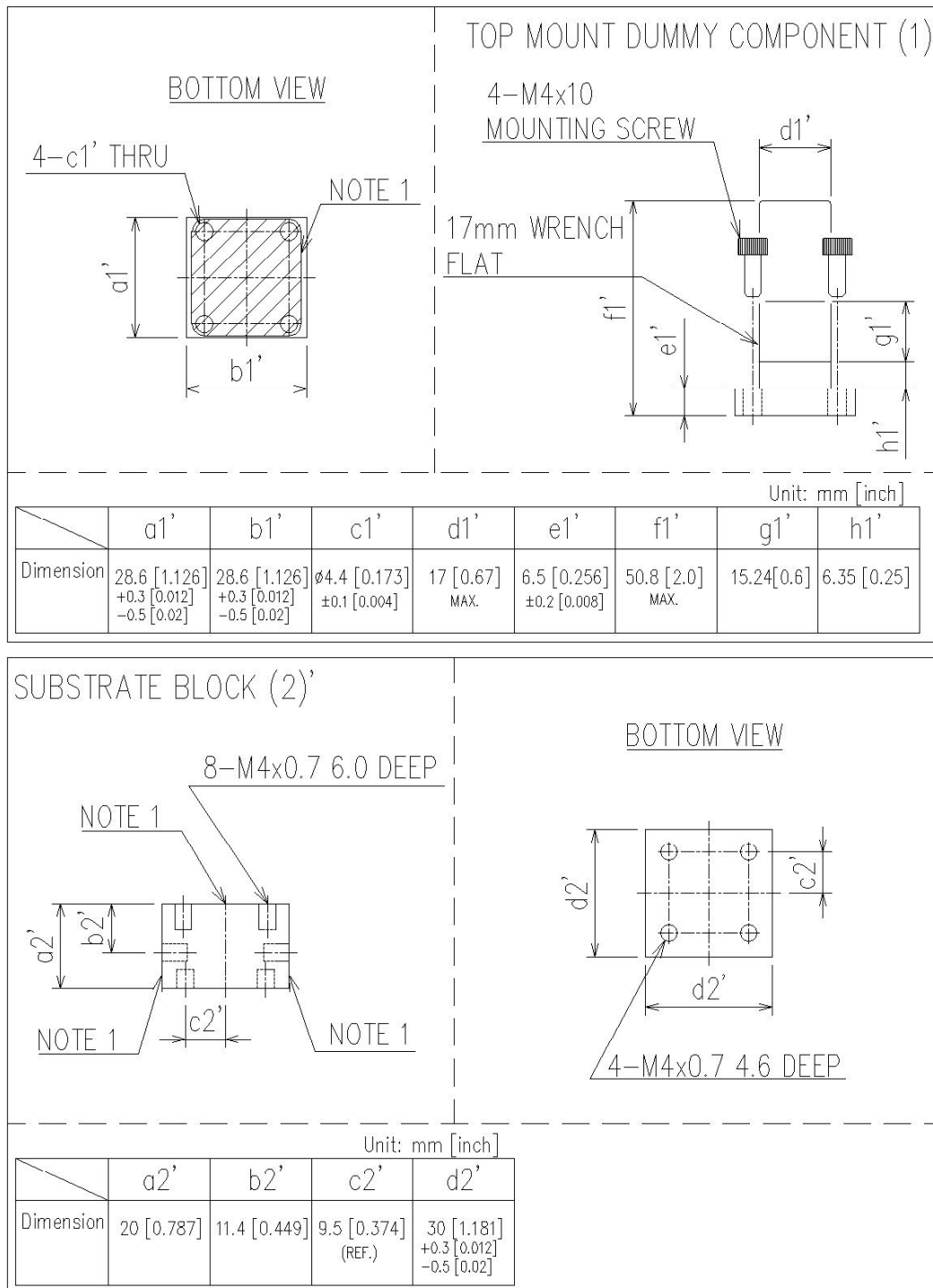
**Figure R1-2b**  
**1. 5" Surface-Mount Performance Testing Fixture**



**Figure R1-2c**  
**1.5" Surface-Mount Performance Testing Fixture**

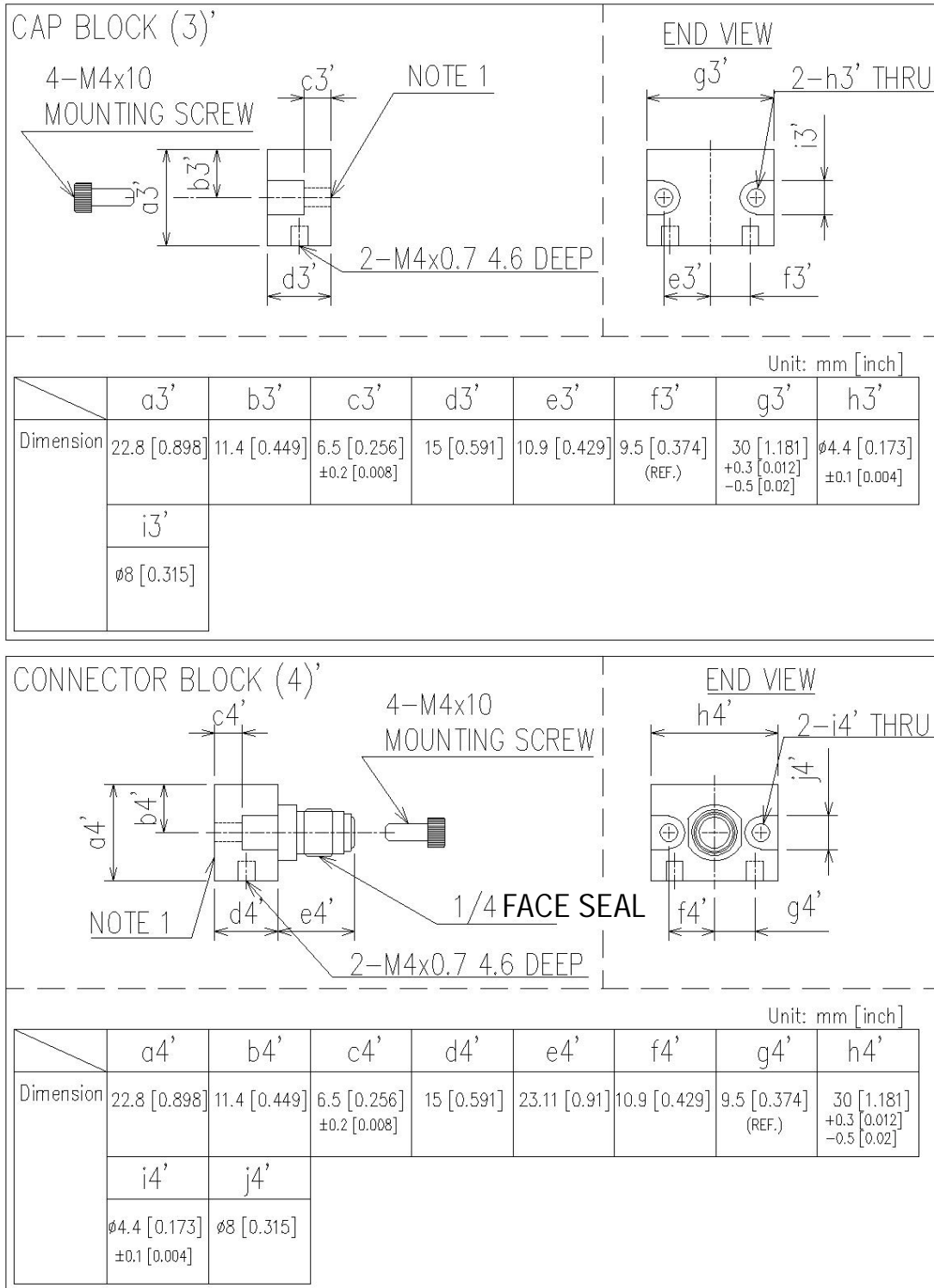


# COMPONENT VIEW; 1.125" VIBRATION/TORQUE TEST FIXTURE



NOTE 1: Leak test port(s) and porting are dependent on seal system/substrate design configuration.  
 NOTE 2: Material of Construction: 316SS.  
 NOTE 3: Drawing scale is not specified.

**Figure R1-3a**  
**1.125" Surface-Mount Performance Testing Fixture**

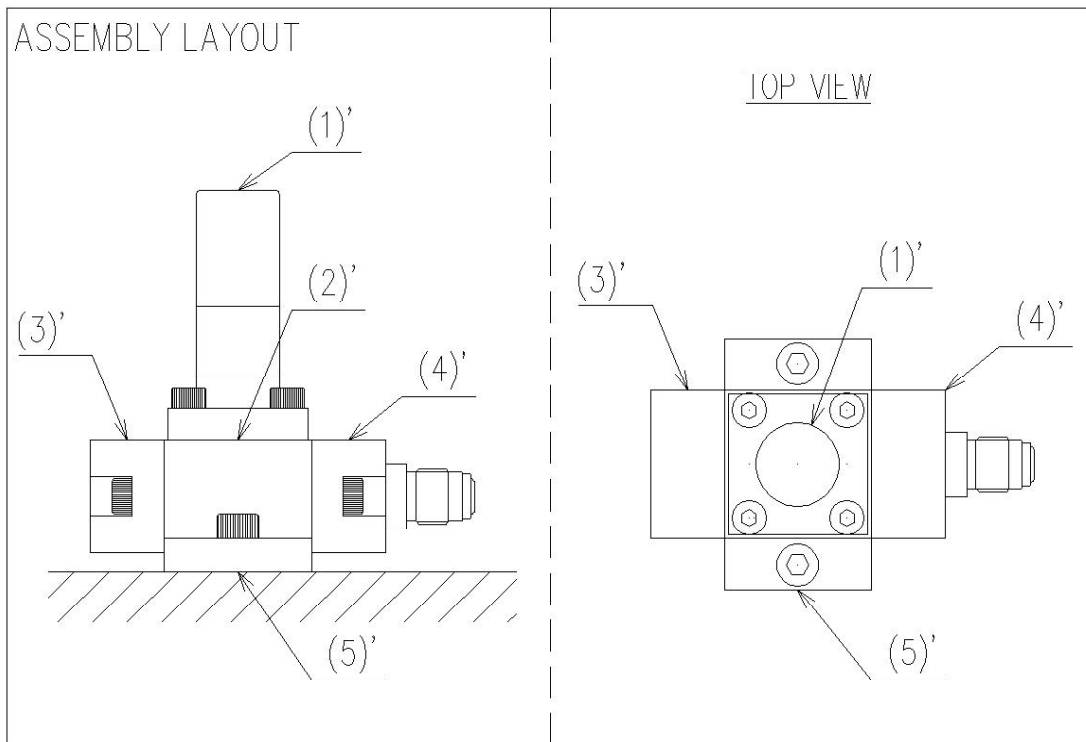
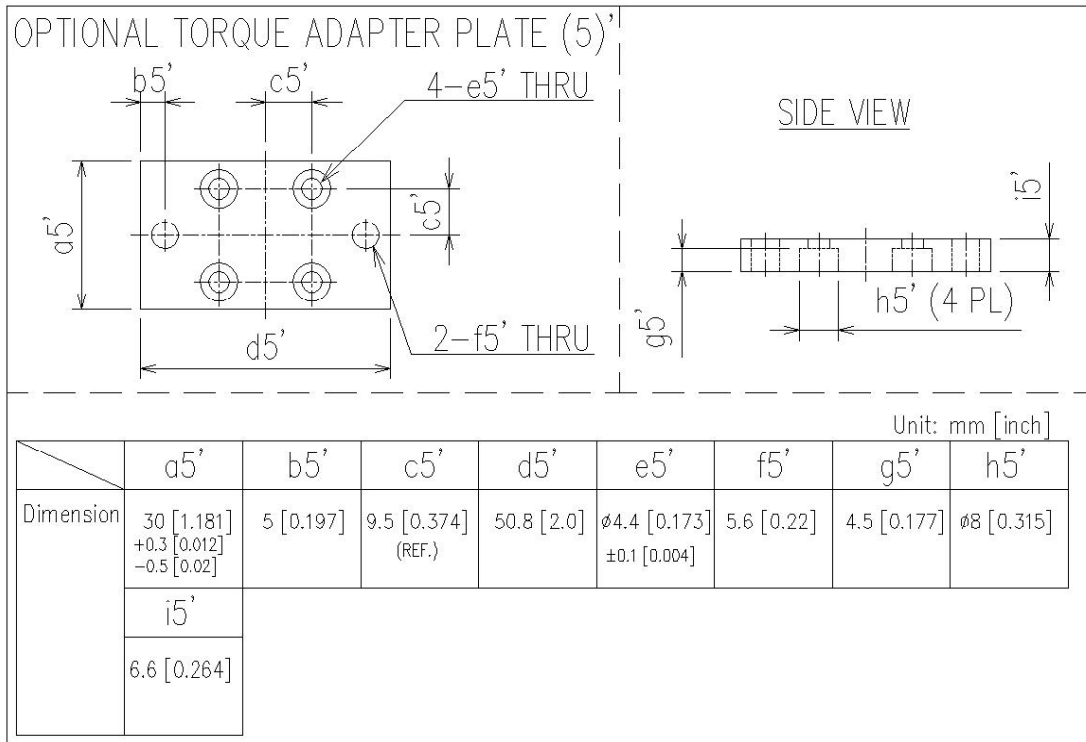


NOTE 1: Leak test port(s) and porting are dependent on seal system/substrate design configuration.

NOTE 2: Material of Construction: 316SS.

NOTE 3: Drawing scale is not specified.

**Figure R1-3b**  
**1.125" Surface-Mount Performance Testing Fixture**



NOTE 1: Leak test port(s) and porting are dependent on seal system/substrate design configuration.

NOTE 2: Material of Construction: 316SS.

NOTE 3: Drawing scale is not specified.

**Figure R1-3c**  
**1.125\"**



**NOTICE:** SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacture' s instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

By publications of this standard, Semiconductor Equipment and Materials International (SEMI) takes no position respecting the validity of any patent rights or copyrights asserted in connection with any items mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights are entirely their own responsibility.

# SEMI F75-1102

## GUIDE FOR QUALITY MONITORING OF ULTRAPURE WATER USED IN SEMICONDUCTOR MANUFACTURING

This guide was technically approved by the Global Facilities Committee and is the direct responsibility of the North American Facilities Committee. Current edition approved by the North American Regional Standards Committee on August 29, 2002. Initially available at [www.semi.org](http://www.semi.org) September 2002; to be published November 2002.

### 1 Purpose

1.1 These guides provide recommendations for facility engineers and other manufacturing and quality professionals who may be responsible for establishing programs to monitor and control the quality of their ultrapure water (UPW) systems through to point-of-use (POU). These guides may be used to help determine the parameters that should be monitored for UPW that is produced, distributed and used throughout the manufacturing facility, and the frequency and location of testing.

NOTE 1: These suggested guides are published as technical information and are intended for informational purposes only.

### 2 Scope

2.1 UPW is used extensively in the production of semiconductor devices for all wet processing steps. Ultrapure water systems need to be tested and monitored to ensure that the UPW being produced matches the specifications established by the manufacturing process. The purity of the UPW may affect device yield unless a wide range of parameters is closely controlled at the point of distribution (POD). Semiconductor devices are currently being designed with smaller linewidths ( $< 0.13\text{--}0.18\text{ }\mu\text{m}$ ) and are more susceptible to low level impurities.

2.2 UPW systems are monitored for continuous performance for desired and achievable levels of quality. Action limits are generally set to determine when system performance data warrants that corrective action is needed. Table 1 *Parameters and Range of Performance* in SEMI F63 may be a useful reference for establishing quality levels.

2.3 In more critical processes, the quality of the UPW also needs to be monitored at the POU where the UPW is in contact with the wafer. The quality of the UPW should not be expected to be identical to the quality of the UPW being produced at final filter (FF), which is not subject to conditions within the tool or distribution system.

2.4 These guides logically follow the series of SEMI guides developed for UPW, which include a standard defining the performance of a UPW system, and a

standard defining the quality of UPW (see reference section). The *Schematic of a Typical Ultrapure Water System* in Figure 1 of SEMI F61 may be a useful reference for determining sampling points.

2.5 Guides are provided concerning the frequency and location of sampling for those parameters that are not available from on-line analyzers. Frequency of sampling should be based on the specifications set by manufacturing for the quality of the POD UPW, the number and locations of on-line analyzers, the stability of the incoming feed water to the system, and the historical performance of the UPW system over time.

2.6 These guides may also be used to establish process control criteria for the incoming feedwater, performance of UPW system components and POU rinse baths.

2.7 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 or regulatory limitations prior to use.

### 3 Limitations

3.1 These guides have been developed with consideration of various other sources deemed to be relevant for this purpose. However this guide was not intended to be identical to or consistent with any other industry document or standard.

3.2 Monitoring and testing of recycled or reclaimed water systems are not specifically addressed in these guides. Additional test parameters, such as quantification and identification of organic species in reclaim water, should be added to monitoring programs for reclaim and reuse waters.

3.3 Measuring the purity of UPW can prove challenging. Many on-line instruments (sodium analyzers, TOC analyzers, silica analyzers, optical particle counters, non-volatile residue analyzers) provide very low limits of detection but may not be capable of being calibrated in the range of detection or may have very poor accuracy at low levels. On-line analyzers should primarily be used for trend analysis. Many tests can still be performed more accurately and reproducibly in a laboratory environment but taking

batch samples can prove time-consuming and be prone to sampling error.

## 4 Referenced Standards

### 4.1 SEMI Standards

SEMI F61 — Guide for Ultrapure Water System Used in Semiconductor Processing

SEMI F63 — Guidelines for Ultrapure Water Used in Semiconductor Processing

NOTE 2: As listed or revised, all documents cited shall be the latest publications of adopted standards.

## 5 Terminology

### 5.1 Abbreviations and Acronyms

NOTE 3: All other abbreviations and acronyms are defined in the SEMI Compilation of Abbreviations and Acronyms available at web site, <http://www.semi.org/>.

5.1.1 *CFU* — Colony-Forming Units

5.1.2 *DO* — Dissolved Oxygen

5.1.3 *EDI* — Electrodionization

5.1.4 *EDX* — Energy Dispersive X-Ray Microanalysis

5.1.5 *FF* — Final Filter

5.1.6 *IC* — Ion Chromatography

5.1.7 *NDIR* — Non-Dispersive Infrared Analysis

5.1.8 *OPC* — Optical Particle Counters

5.1.9 *POD* — Point Of Distribution

5.1.10 *RO* — Reverse Osmosis

5.1.11 *TDS* — Total Dissolved Solid

5.1.12 *THM* — Trihalomethanes

## 6 Units

6.1 Parts per million (ppm) is equivalent to  $\mu\text{g/mL}$  or  $\text{mg/L}$ .

6.2 Parts per billion (ppb) is equivalent to  $\text{ng/mL}$  or  $\mu\text{g/L}$ .

6.3 Parts per trillion (ppt) is equivalent to  $\text{pg/mL}$  or  $\text{ng/L}$ .

## 7 Use of the Guides

7.1 Monitoring programs should reflect the age and complexity of the UPW equipment and the needs of the manufacturing process.

7.2 The quality of UPW at the POU may be negatively impacted by 1.) contamination sources within a tool, 2.)

the design of the tool, 3.) the materials of construction of the tool and piping distribution, and 4.) contamination loading in the bath from build-up after multiple rinse cycles.

7.3 Sampling methods and contamination control are of paramount importance when attempting to measure the listed parameters at very low levels of sensitivity.

7.4 The quality of the data measured may depend on which testing methods and calibration techniques are used. Consequently, trends observed in the values may be more meaningful than absolute values, especially for certain on-line monitors calibrated for ultrasensitive detection. In addition, online and offline measurements may not correlate depending on the measurement technique and level of sensitivity of the measurement.

## 8 Tests For Monitoring UPW Production and Distribution

NOTE 4: Since SEMI Guides do not require analytical data or methods to support them, the recommendation of specific analytical methods are only for informational purposes. Alternative methods may also be applicable.

NOTE 5: See Table 1 at the end of this document for summary of recommended testing frequency and sampling points.

### 8.1 Resistivity (*megohm-centimeters*)

8.1.1 Resistivity (the inverse of conductivity) is a general measure of ionic activity and is measured by an on-line meter. The resistivity of UPW should be approximately  $18.2 \text{ m}\Omega\text{cm}$  depending on the resolution of the instrument.

NOTE 6:  $18.25 \text{ m}\Omega\text{cm}$  is the theoretical upper limit for pure water at  $25^\circ\text{C}$ .

### 8.2 Total Oxidizable (Organic) Carbon (TOC) (*ppb*)

#### 8.2.1 Carbon Sources in Water Supplies

8.2.1.1 Incoming feed water contains both inorganic and organic carbon. Inorganic carbon as dissolved carbon dioxide ( $\text{CO}_2$ ), bicarbonate, and carbonate can be present at high ppm levels according to the geology of the water supply. Organic carbon reflects biological input and man-made contaminants such as oils, pesticides, and fertilizers. Ground and well waters normally have significantly lower organic content than surface waters. To remove the majority of organics, most UPW systems employ reverse osmosis; anion exchange resin and ultraviolet (UV) destruction are also employed to remove organics. Some volatile organics, such as trihalomethanes (THM), may be controlled by the use of vacuum degasification. The control of organics is essential for preventing organic fouling and maintaining resin beds at high efficiency. In addition,

reduced organic matter will limit the nutrients available for bacteria growth and thus biofilm development. Increasing TOC values at the FF can indicate degradation and/or fouling of system components, contamination from routine operational maintenance, or return contamination from the factory. Changes in the incoming feed water due to seasonal or extreme weather conditions can also cause increases in TOC readings.

#### 8.2.2 *Method of TOC Measurement for Source Water*

8.2.2.1 There are various methods for measuring TOC, and several TOC analyzers are available. TOC measurement generally involves the oxidation of organic materials by means of temperature, UV radiation, and/or chemicals. The CO<sub>2</sub> produced by these reactions can then be measured by non-dispersive infrared analysis (NDIR) or conductivity (resistivity) differential. The exact method utilized will vary depending on the TOC instrumentation employed and the method may also affect the TOC readings as different methods have different recoveries of various organics. Some instruments will also require the use of a carrier gas such as air or nitrogen, while others measure TOC directly.

#### 8.2.3 *Monitoring TOC in the UPW System*

8.2.3.1 TOC is a useful test to measure the organic removal effectiveness of the UPW system components including Carbon, RO, Degasification, and Ion Exchange. Suggested measurement points are city feed, pre and post RO membrane, and final filter, with a minimum frequency that reflects possible changes in organics from the feed source or UPW components. After the RO, TOC drops from low ppm levels in the source water to mid ppb range, and to single digit ppb levels after the mixed resin beds and UV TOC reduction units.

#### 8.2.4 *Monitoring TOC at the POU*

8.2.4.1 TOC may be measured at the point-of-use to determine quality changes from the distribution system and the manufacturing tool. Short wavelength UV (185 nm) is capable of breaking up residual organics into charged organic molecules. TOC which survives to the point of use in a UPW system are typically either “light” molecules or small fragments of larger molecules such as acetate and formate. While low TOC means that the UPW system is working effectively to eliminate the source water organic load, this test is not an accurate measure of sterility of a UPW system. In addition, TOC levels at POU can also reflect carryover from chemical baths and contaminants in cleanroom air.

#### 8.3 *Dissolved Oxygen (ppb)*

8.3.1 Dissolved oxygen (DO) content can be

controlled, if desired. Rinsing hydrogen passivated silicon wafer surfaces with high DO UPW can result in etching of the silicon by the oxygenated UPW and loss of control of gate oxide thickness.

#### 8.4 *Particulate Matter (Particles/L)*

##### 8.4.1 *Sources of Particles in Ultrapure Water Supplies*

8.4.1.1 Particles that adhere to wafer surfaces at each step of the integrated circuit device manufacture may impair the application of thin-films and photolithographic substances, and ultimately cause discrete and integrated transistors to fail because of resultant physical imperfections. Particles originate in the incoming city water and may also be generated from degradation of the UPW system components or operational activities. Incoming source water contains a high level of particles and is initially filtered by prefilters and mixed media in order to remove gross physical turbidity in the micron range.

8.4.1.2 Following reverse osmosis, source water particles have been greatly reduced, as the RO membranes also reject particles. It is essential to minimize the particle load to the RO to prevent membrane fouling and premature or frequent RO cleanings. From the RO outlet forward in the system, the particle load in the UPW originates from system components. Particle sources can be RO membrane modules, piping components, valves and other similar control devices, tank linings, resin fines and other sources where materials are undergoing wear or degradation and are shedding particles.

8.4.1.3 After the ion-exchange resin beds, increasingly smaller dimension filters are used in the range 0.2 to 1.0 microns to remove residual particles and resin fines. In most ultrahigh purity applications, ultra filtration at 10,000 molecular weight is utilized. In most UPW systems, the presence of significant quantities of sub-micron particles at the final filter may indicate the breakdown of upstream UPW system components. In addition, POU filters on tools can be a source of contamination if not maintained.

##### 8.4.2 *Optical Particle Counters (OPC)*

8.4.2.1 On-line methods using laser optical particle counting technology are recommended for trend analysis and specification compliance. These instruments can monitor particles in the 0.03 to > 0.5 micron range depending on the system component being tested. Particle counting using an OPC requires a good sample port connection to eliminate false counts. Good counter maintenance, including annual calibration and replacement of tubing and fittings is critical especially if monitoring particles ≤ 0.05 microns. In order to monitor trend analysis with good statistical

process control (SPC), the number of particles per sample interval should be sufficient for good statistics (e.g. > 20 particles per sample interval). For example, to obtain a coefficient of variation (standard deviation/mean) of 10%, it is necessary that at least 100 particles greater than the control be counted.

#### 8.4.3 *Scanning Electron Microscopy (SEM) Analysis or Direct Count Analysis of Particles and Bacteria*

8.4.3.1 SEM is used to analyze particles and bacteria within the UPW system. Three major aspects of the SEM method are the enumeration of particles, enumeration of bacteria and the determination of the elemental composition of the captured particles. Depending on the particulate level in the UPW, a large volume of UPW is filtered on-line, typically at the final filter. Due to particles created by the act of sampling and the preexisting particles found normally on a new filter prior to use, a volume of over 1000 liters may be required to get a statistically valid result. In the laboratory, the filter is removed from the filter holder and placed in the vacuum chamber of a SEM instrument. A focused beam of electrons is scanned across the filter, systematically allowing the size, shape and distribution of the particles to be measured. The electron microscope is capable of extremely high power detection (10,000× magnification), and the counting process may be automated. Bacteria and particles in the 0.05 to > 0.5 micron range are detected and counted.

8.4.3.2 When SEM particle capture filters are installed at critical areas in the UPW system on a continuous basis, they may be utilized to determine the source and nature of intermittent particle excursions.

#### 8.4.4 *Identification of Particles*

8.4.4.1 As part of the SEM analysis, an Energy Dispersive X-ray Beam (EDX) may be applied to a particle, which produces a spectral output of the composition of the particle. EDX results allow the basic classification of organic and inorganic components. Elemental compositions are provided that can be used to trace the source of particulates. For example, a particle having Cr and Fe peaks is probably from a stainless steel source, while one having C and F peaks is most probably composed of fluoropolymer materials.

### 8.5 *Bacteria*

#### 8.5.1 *Sources of Bacteria in Water Supplies*

8.5.1.1 Incoming city water supplies and UPW system components are all potential sources of biological contaminants if not monitored and controlled. Viable (live) counts are usually non-detectable or very low in the city water due to chlorination. Chlorine removal and low flow areas in the components are the main catalysts

for rapid biological growth in an UPW system. Fouling of RO membranes and ion-exchange resins may occur, requiring costly remediation. Seasonal variations may greatly change the bacterial count of source water.

8.5.1.2 The oligotrophic nature of the UPW causes the bacteria to become hydrophobic which creates a thermodynamic adhesion to the wall. The bacteria, once adhered to the wall have a higher degree of probability of having the nutrients required for growth brought by them than the planktonic bacteria in the system. This process initiates the creation of a biofilm. While some system owners employ sanitization programs in hopes of safeguarding against microbial activity, biofilm can be prove resistant and may permanently coat the inaccessible surfaces of valves and dead-legs. Despite the fact that UPW systems are designed to be hostile to most bacterial species, the formation of biofilm on filters, in membranes, and in ion-exchange resins is widespread and periodic flourishing of bacterial colonies can be a costly long-term problem. Once in the tool, live bacteria can multiply further particularly in low flow area and pose an ever greater threat.

#### 8.5.2 *Viable Bacteria by Culture*

8.5.2.1 Replicate samples are collected at each sampling point in sterile containers, passed through special sterile filters, and then dosed with a growth medium. In an incubator, bacteria are cultured and grow to form colonies. The bacteria colonies are counted under low power magnification after a specified interval and at a specified temperature. Different methods will enumerate different results. Results may vary depending upon factors such as sample volume, growth media, incubation time and temperature, and enumeration methods. It may be necessary to evaluate different methods to determine the best recovery for a specific UPW system. Bacteria results are reported as colony-forming units (CFU) per unit volume (e.g. 100 mL or 1000 mL). For very low bacteria UPW systems, larger sized samples may be collected to provide lower detection limits. Larger volumes can be filtered using this method, making the bacterial counts more statistically significant.

#### 8.5.3 *Total Bacteria by Epifluorescence*

8.5.3.1 After collection, samples are filtered onto polycarbonate membranes, which are then stained with dyes that cause biological materials to fluoresce under ultraviolet light. A high power microscope is employed to visually identify bacteria, both live and dead, and a counting method is used to give statistical accuracy for the sample size. This test provides accurate information about actual bacterial content (both viable and non-viable) of the UPW system, rather than an



estimate of bacteria that is capable of being cultured during a growth opportunity (the culture method). On line samples may also be obtained utilizing a method similar to that of the SEM particle capture method. Larger volumes can be filtered using this method making the bacterial counts more statistically significant. The culture method may also be performed concurrently to determine if an increase in levels is due to live or dead bacteria.

#### 8.5.4 *Total Viable Organisms by Scan RDI*

8.5.4.1 Scan RDI is a semi-automated method of measuring total viability counts. The method is able to detect a single cell based on direct measurements of cell activity and includes bacteria and other live organisms that may be present in biofilm.

8.5.4.2 The sample is filtered through a membrane and a counterstain is added to minimize background fluorescence. Viable organisms are labeled using a nonfluorescent substrate that diffuses across the cell membrane. The labeling differentiates between viable and dead cells based on the presence or absence of esterase activity and intact cell membranes. Only viable cell with membrane activity have the ability to perform this cleavage and retain the fluorescent label. No cell growth is required. The membrane is then scanned by a solid-phase laser-scanning cytometer. The cells are detected, counted and verified within minutes.

### 8.6 *Silica*

#### 8.6.1 *Forms of Silica in UPW Systems*

8.6.1.1 Silica exists primarily in two forms as silicates and as polymeric silica. Silicates are referred to as dissolved silica and are reactive to molybdate using the heteropoly method. The polymeric forms are called colloidal silica or particulate silica and are not measured directly by on-line analyzers. As pH in a UPW system is lowered, silicates polymerize to form colloidal silica, which range in size from a few nanometers to several tenths of a micron.

8.6.1.2 As with TOC, RO membranes are responsible for the gross rejection of silica in incoming waters. Within the UPW system, dissolved silica is further removed in the anionic ion exchange resin beads. However, dissolved silica is only weakly attracted to the anionic resins, and is one of the first species to be liberated when the resin beds approach exhaustion. After anion resins are exhausted, increasingly high levels of silica are released into the UPW stream. Since dissolved silica is only weakly negatively charged, the on-line resistivity meter is not sensitive enough to detect silica leakage prior to total resin bed exhaustion, i.e. breakthrough. It is very important to detect this onset of silica breakthrough before a drop in resistivity. If

this does occur, then  $\text{Cl}^-$  and other monovalent ions may pass from the resin to the point of use. This would also be accompanied by a decrease in the resistivity from the resin beds. This occurs because ions that are stored on the resin beds over a period of time become liberated as their exchange sites are taken over by other higher-affinity ions (e.g. nitrate, sulfate).

#### 8.6.2 *Dissolved Silica Techniques*

8.6.2.1 For the measurement of dissolved silica, a colorimetry heteropoly blue method is used. Dissolved silica reacts with the molybdate reagent at acidic pH, and forms a yellow complex in direct proportion to concentration. A reducing agent is added to the prepared sample, causing a blue color to develop. This color change can be quantified by a sensitive spectrophotometer for ppb to sub-ppb levels of dissolved silica. On-line instruments provide continuous monitoring of UPW systems. Ion Chromatography may also be used to measure dissolved silica.

#### 8.6.3 *Total Silica Technique*

8.6.3.1 Since all forms of silica are not reactive in the heteropoly method, the quantity of total silica in UPW must be verified independently. Techniques such as ICP-MS are capable of quantifying the silicon present to sub ppb levels after pre-concentration. The method detection limit of total silica obtained by this technique is in the range of 0.5 ppb.

#### 8.6.4 *Measurement Methods For Total Silica*

8.6.4.1 Total Silica may be measured by Graphite Furnace Atomic Absorption Spectroscopy (GFAAS), Inductively Coupled Plasma Atomic-Emission Spectroscopy (ICP-AES) or Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) at ppb levels.

#### 8.6.5 *Colloidal Silica*

8.6.5.1 The difference between total silica and dissolved silica is assumed to be represented by the colloidal form of silica. Colloidal silica is partially removed by the RO membrane, however, some hot UPW feed streams may carry a higher concentration of colloidal silica. Colloidal silica cannot be detected by on-line resistivity or dissolved silica testing, but may be detected as a component of non-volatile residue.

#### 8.7 *Non-Volatile Residue (NVR)*

8.7.1 Non-volatile residue in UPW primarily consists of dissolved inorganic material. In the final polished UPW the most likely dissolved inorganic material is silica in both the dissolved and colloidal form. The primary source of this breakthrough silica is the anion resin bed as it approaches exhaustion. Sub-ppb levels of dissolved silica can be detected as non-volatile

residue. There is also evidence that as an ion-exchange bed approaches exhaustion, previously removed dissolved silica can be subsequently released as colloidal silica if the pH in the ion-exchange bed is favorable. There is currently no specific measurement of on-line colloidal silica, but detecting it as a component of non-volatile residue can be beneficial.

#### 8.7.2 Method of Measurement for NVR

8.7.2.1 The on-line measurement of nonvolatile residue is based on the principle of determining “residue after evaporation” of atomized ultrapure water droplets. Droplets evaporate in a fraction of a second leaving an ultrafine nonvolatile residue particle agglomerate a few nanometers in diameter; the size of which is related to the amount of residue impurity originally present in the ultrapure water. A condensation particle counter then monitors the concentration of ultrafine nonvolatile residue particles. The measurement range of this technique is 0.01 parts per billion to 20 parts per million.

#### 8.8 Ions

##### 8.8.1 Mass Balance and Ionic Removal in UPW Systems

8.8.1.1 The ionic composition of natural waters is in a dynamic equilibrium (mass balance) with the geological source. The total dissolved solid (TDS) content is composed of negatively charged (anionic) and positively charged (cationic) species that can be analyzed by Ion Chromatography (IC). RO membranes, ion-exchange resins, and electronic deionization (EDI) are used to remove ionic species from water. The UPW resin beds need to be monitored because each unit has a fixed capacity to remove ions over time. Weakly bound singly charged (monovalent) ions such as  $\text{Na}^+$  and  $\text{Cl}^-$  are continually displaced in favor of more highly charged doubly charged (divalent) ions such as  $\text{SO}_4^{2-}$  and  $\text{Ca}^{2+}$  throughout the life of the resin. When ionic breakthrough occurs, the weakly bound ions are liberated in excess, and resin beds must be regenerated.

##### 8.8.2 Ion Chromatography Techniques

8.8.2.1 Ionic (anions and cations) removal in UPW systems can be quantified at very sensitive levels using the technique of ion chromatography. During IC analysis, a UPW sample is injected into an analytical column packed with a special ion-exchange resin. The ions from the samples are eluted (washed) down the column by a suitable eluent. Depending on their binding energy and sizes, the ions move down the column at different speeds. In time, all of the ions are separated and measured by a conductivity detector. External calibration of a sensitive conductivity detector across a wide range of concentrations allows accurate

quantification of anion and cations. The anions and cations determined by IC are fluoride ( $\text{F}^-$ ), chloride ( $\text{Cl}^-$ ), nitrite ( $\text{NO}_2^-$ ), bromide ( $\text{Br}^-$ ), nitrate ( $\text{NO}_3^-$ ), sulfate ( $\text{SO}_4^{2-}$ ), phosphate ( $\text{PO}_4^{3-}$ ) and lithium ( $\text{Li}^+$ ), sodium ( $\text{Na}^+$ ), ammonium ( $\text{NH}_4^+$ ), potassium ( $\text{K}^+$ ), magnesium ( $\text{Mg}^{2+}$ ), calcium ( $\text{Ca}^{2+}$ ) respectively.

#### 8.8.3 Ions Critical in Semiconductor Processing

8.8.3.1 Certain types of haze formation on wafer surfaces could be caused by high concentrations of anions and cations on the silicon surface as residue. There could be other causes for haze formations, therefore, testing of anions and cations at the POU in the rinse bath may be important to identify and prevent this source of contamination.

#### 8.9 Metals

##### 8.9.1 Measurement Methods

8.9.1.1 Up to 68 metals may be determined by instruments such as Graphite Furnace Atomic Absorption Spectroscopy (GFAAS), Inductively Coupled Plasma Atomic-Emission Spectroscopy (ICP-AES) or Inductively Coupled Plasma-Mass Spectrometry (ICP-MS).

##### 8.9.2 Sources of Trace Metals

8.9.2.1 Trace metal concentrations in feed waters vary over orders of magnitude according to the geology of source rocks and the residence time of water in aquifers and surface systems. Surface water supplies are often mixed by the local municipal services, and seasonal variations are also introduced as ground water and well waters are brought in and out of service. Consequently, periodic analysis of metals in incoming feedwater and at key points of the UPW system including after the final filter is essential so that significant changes in performance may be corrected early in the process of deionization.

##### 8.9.3 Boron

8.9.3.1 Boron is a trace element that, like silica, is weakly attracted to anion resins. Both elements serve as a flag for the onset of ionic breakthrough through the anion exchange resin. A rapid and systematic rise in the boron content immediately after the resin bed is typical when ion-exchange resins approach exhaustion in the UPW system. Silica and boron breakthrough rates and each element's selectivity in the ion exchange process is a matter of ongoing study. The safest route is to measure both, but at least to focus on the one most prevalent in the city water feed stream.

##### 8.9.4 Contaminants from the Distribution System

8.9.4.1 Certain heavy elements such as Zn, Mn, Ni, Cu, Cr, and Pb may be present as trace impurities in plastic

(PFA and PVDF) components and are continuously leached into the UPW streams. Plastic lined metal components such as pumps, storage tanks, and other UPW processing vessels may develop pinholes, thereby leaching out heavy metal impurities into the UPW system.

#### 8.9.5 *Contaminants from Feed Water*

8.9.5.1 The alkali metals (Na, K), alkali earths (Mg, Ca) and Fe comprise the bulk elements of most natural waters, and play a significant role in bulk water chemistry including hardness, alkalinity and total dissolved solids. Of these elements, sodium is the most abundant and is present in final filter UPW at sub-ppb level. Highly charged metals (Al, Fe, and Mn) have the potential to coagulate and foul RO membranes, and Ba and Sr can cause scaling of the RO and ion exchange resins.

#### 8.9.6 *Priority Metals in Semiconductor Processing*

8.9.6.1 Certain processing steps expose a wafer to contamination from UPW in a unique way. Metals from incoming materials (e.g., chemicals, wafers, etc.), deteriorating wafer processing equipment, and new processes and materials for deposited circuit layers can lead to cross contamination and high concentrations of metals “gettering” on the silicon wafer surface. Testing for metals in the UPW at POU (e.g., the rinse bath) may be important in identifying and preventing sources of metal contamination on the wafer. Certain other elements, calcium, magnesium and aluminum, have an increased ability to contaminate the oxides of wafers. There may be other metals that, depending on the semiconductor process, can also act as harmful contaminants.

#### 8.9.7 *Radioactive Elements*

8.9.7.1 Radioactive elements such as uranium and thorium can cause soft X-ray damage to devices when present at ppt levels. These elements are detectable in a full ICP-MS scan.

## 9 **Typical Monitoring Programs**

### 9.1 *On-Line Monitoring*

9.1.1 On-line monitors for UPW are readily available from a variety of manufacturers. Before selecting an instrument for on-line monitoring an evaluation should be made about 1.) sensitivity of measurements, 2.) accuracy of the measurements, 3.) reproducibility of the measurements, 4.) calibration protocols for the instrument, 5.) zero count for the instrument (particle counters) and 6.) instrument cost and reliability.

9.1.2 If on-line monitors are not available at critical sampling points, or the sensitivity of the on-line monitor is not sufficient to accurately measure at the required levels, grab samples may be taken and sent to a qualified laboratory for equivalent testing. This off-line sampling and testing requires special apparatus and trained personnel. A clear understanding of the limits of each method is required before its application.

9.1.3 All on-line monitoring requires clean and tight sample port connections in order to eliminate false counts.

9.2 Testing at critical sampling points, such as point-of-use, requires special sampling apparatus and trained personnel. The enclosures and mini-environments around wet benches that are designed to prevent accidental access and to isolate human contamination from the wafer can make it difficult to sample the bath. However, contamination-free sampling can be done in a safe way using high purity fluoropolymer apparatus and trained qualified personnel.

9.3 The utilization of unbiased, independent and experienced personnel in obtaining data at critical sampling sites with the UPW system where on-line instrumentation is not available is essential in determining the overall performance of the UPW system.

9.4 See Table 1, “UPW System Test Parameters, Sampling Points, and Recommended Monitoring Frequency for Assuring Good UPW System Operation” for sample point and monitoring frequency recommendations.

**Table 1 UPW System Test Parameters, Sampling Points and Recommended Monitoring Frequency for Assuring Good UPW System Operation**

<i>Type of Water</i>	<i>Source Incoming Feed pre RO filters</i>	<i>Pre RO Post RO prefilters</i>	<i>UPW Post RO</i>	<i>UPW Post Ion Exchange</i>	<i>UPW Post Final Filter</i>	<i>UPW Return Loop</i>	<i>UPW POU Tools (Recommended)</i>
Resistivity	-	Continuous (Conductivity)	Continuous (Conductivity)	Continuous (Conductivity)	Continuous (Conductivity)	Continuous (Conductivity)	-
TOC	Bi-Weekly	Bi-Weekly	Continuous	Continuous	Continuous	Continuous	Bi-Weekly
Particles OPC	Monthly	Monthly	Monthly	Continuous	Continuous	Continuous	Monthly
Particle SEM	-	-	-	Bi-monthly	Monthly	Bi-monthly	Monthly
Bacteria	Biweekly	Biweekly	Biweekly	Biweekly	Weekly	Weekly	Bi-weekly
Dissolved Silica	Monthly	Monthly	Monthly	Continuous	Continuous	Continuous	Monthly
Total Silica	Monthly	Monthly	Monthly	Bi-weekly	Weekly	Weekly	Bi-weekly
NVR	-	-	-	Continuous	Continuous	Continuous	-
Sodium	-	-	-	Continuous	Continuous	Continuous	-
Boron	Monthly	Monthly	Monthly	Quarterly	Weekly	-	-
Anions & Cations	Monthly	Monthly	Monthly	Bi-weekly	Bi-weekly	Bi-weekly	-
Critical Ions <sup>1</sup>	-	-	-	-	-	-	Bi-weekly
Trace Metals-Critical <sup>2</sup> Elements	-	-	-	Bi-weekly	-	-	Bi-weekly
Trace Metals-Full Scan	Quarterly	Quarterly	Quarterly	Quarterly	Monthly	Monthly	-

<sup>1</sup> Critical Ions include Chloride, Fluoride, and Ammonium.

<sup>2</sup> Critical Elements include Al, Ca, Cr, Cu, Fe, Mg, Ni, Na, and Zn

NOTE 7: The frequency noted for these parameters is not intended to assure against excursions due to improper maintenance procedures or other external means of system contamination.

**NOTICE:** SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature respecting any materials mentioned herein. These standards are subject to change without notice.

The user's attention is called to the possibility that compliance with this standard may require use of copyrighted material or of an invention covered by patent rights. By publication of this standard, SEMI takes no position respecting the validity of any patent rights or copyrights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights, are entirely their own responsibility.

# SEMI F76-0303

## TEST METHOD FOR EVALUATION OF PARTICLE CONTRIBUTION FROM GAS SYSTEM COMPONENTS EXPOSED TO CORROSIVE GAS

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 November 22, 2002. Initially available at [www.semi.org](http://www.semi.org) December 2002; to be published March 2003.

### 1 Purpose

1.1 This is a test method to compare gas handling components for potential particle generation in corrosive gas service. It is intended as a practical means of generating performance data for a group of components to be compared in a selection process.

### 2 Scope

2.1 This method applies to valves, particle filters, and low pressure regulators.

**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 and health practices and determine the applicability of regulatory or other limitations prior to use.

### 3 Limitations

3.1 This method is not intended to provide insight into fundamental corrosion mechanisms.

3.2 Since the test is to be conducted on fully functional gas system components, it is not designed to determine the suitability of specific materials of construction for corrosive gas service. Such factors as component design, flow path, weld quality, cleanliness of construction and manufacturing methods can impact the results of this test making the material of construction only one of many factors.

3.3 This method is not designed to provide reliability or failure data. It is expected that the data produced will be relative; that is, the data will be most useful when groups of components are compared to each other.

3.4 This method is not designed to evaluate parameters besides particle contribution, for instance leak rate or regulator drift.

3.5 Components to be tested must be capable of withstanding the bake temperature of 100°C for filters and 70°C for valves and regulators.

3.6 This test applies to HCl which is a reducing environment. Results may not be indicative of

performance in corrosive oxidizing environments such as Cl<sub>2</sub>.

### 4 Referenced Standards

#### 4.1 SEMI Standards

SEMI C3.49 — Standard for Bulk Nitrogen (N<sub>2</sub>), 99.99999% Quality

SEMI E66 — Test Method for Determining Particle Contribution by Mass Flow Controllers

#### 4.2 ASTM Standard<sup>1</sup>

ASTM F1394-92 — Standard Test Method for Determination of Particle Contribution from Gas Distribution System Valves.

#### 4.3 SEMATECH Documents<sup>2</sup>

SEMASPEC #93021510A-STD — Test Method for Determination of Particle Contribution by Low Pressure Regulators in Gas Distribution Systems.

SEMASPEC #93021511A — Test Method for Determination of Particle Contribution by Filters in Gas Distribution Systems.

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

### 5 Terminology

#### 5.1 Definitions

5.1.1 *cycle purge* — a procedure which employs alternating cycles of vacuum and pressure for the purpose of evacuating a gas system or a portion of a gas system of impurities or active gases.

5.1.2 *particle filter* — a device that removes small particles from a gas stream with high efficiency.

5.1.3 *point-of-use purifier* — an inline device that removes homogeneous impurities such as moisture and oxygen gases. An integral particle filter is typically included in the same housing to remove particles.

<sup>1</sup> American National Standards Institute, 1819 L Street, NW, Suite 600, Washington, DC 20036, website: [www.ansi.org](http://www.ansi.org)

<sup>2</sup> SEMATECH, 2706 Montopolis Drive, Austin, TX 78741, website: [www.sematech.org](http://www.sematech.org)

5.1.4 *regulator* — a valve designed to reduce a high incoming pressure to a lower outlet pressure by automatically opening to allow flow until a desired, pre-set pressure on the outlet side is reached, and then automatically throttling to stop further pressure increase.

5.1.5 *valve* — a device that controls the flow or pressure of a gas. Valve functions can include shut-off, metering, backflow prevention, and pressure relief.

## 6 Summary of Method

6.1 This test method is designed to provide comparative results for similar components. It is not intended to replicate process condition or actual use conditions. Components will be compared for their particle shedding performance after being exposed to moisture and corrosive gases, in this case hydrogen chloride (HCl) gas. The component will be exposed alternately to corrosive gas and then to moist 100 ppm nitrogen gas several times. The particle shed performance before and after exposure will be compared. The overall test sequence is outlined in Figure 2.

## 7 Apparatus

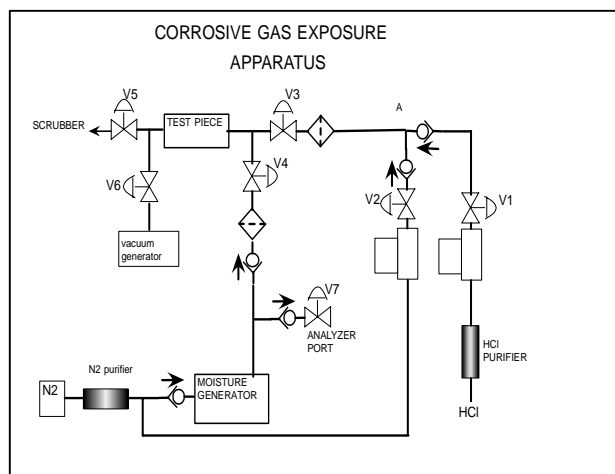
### 7.1 Corrosive Gas Exposure Apparatus

7.1.1 The corrosive gas exposure will be carried out using the apparatus shown in Figure 1.

7.1.2 *Moisture Generator* — Moisture generation is achieved using a permeation-source-type moisture generator. The permeation source should be selected to provide 100 ppm H<sub>2</sub>O in a flow of 500 sccm nitrogen. Records should be kept on the age of the permeation source so that a reliable source of moisture can be maintained.

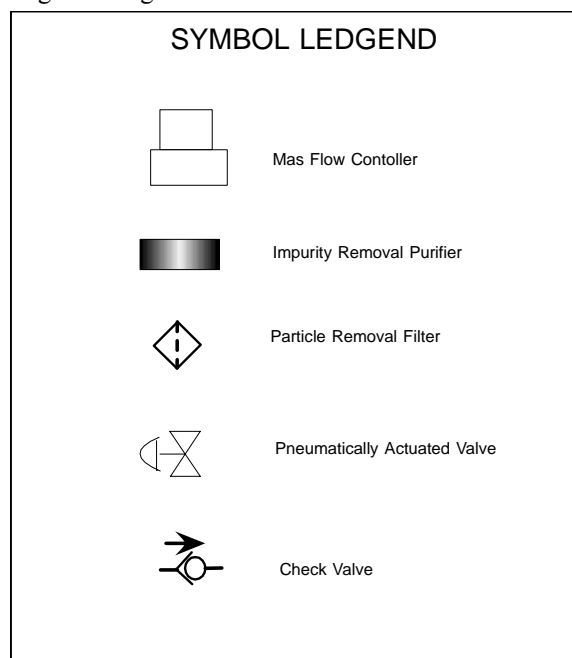
7.1.3 *Vacuum Generator* — A Venturi or similar device capable of producing a rough vacuum of 85000 pascals (25 inches of mercury).

7.1.4 *Point of HCl Introduction* — The point at which gaseous hydrogen chloride HCl and N<sub>2</sub> join the system (point A in Figure 1) should be designed so that the dead volume between the check valves is minimized. The check valves following V1 and V2 should be nearly joined to each other so as to minimize this volume.



**Figure 1**  
**Corrosive Gas Exposure Apparatus**

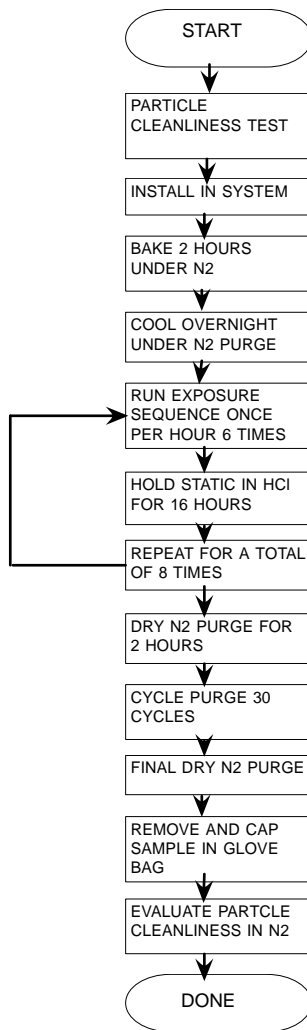
Legend to figure 1



## 8 Reagents and Materials

8.1 *Nitrogen* — Nitrogen with less than 50 ppbv moisture is to be used. This can be accomplished with a point-of-use purifier as shown in Figure 1 or by using 99.99999% quality bulk nitrogen according to SEMI C3.49.

8.2 *Hydrogen Chloride* — Gaseous HCl should contain less than 1 ppmv H<sub>2</sub>O. The HCl purifier shown in Figure 1 is optional unless the source gas contains greater than 1 ppmv H<sub>2</sub>O.



**Figure 2**  
**Test Flow**

## 9 Safety Precautions

9.1 This method involves the use of hazardous materials. The user should be experienced in the handling and use of corrosive specialty gases.

9.2 Safety precautions should include, but are not limited to:

- continuous air monitoring for HCl in the working environment as well as the ventilated enclosure.
- SCBA or other supplied air respiratory protection capable of use in gas exposure levels that may exceed immediately dangerous to life and health (IDLH) levels should be used during cylinder installation and start-up.

- Complete enclosure of the HCl exposure apparatus in a forced-ventilation cabinet.
- The user should consult the safety, health and environmental personnel at their site for advice on appropriate personal protective equipment, monitoring and ventilation requirements.

## 10 Test Specimen

10.1 *Starting Condition* — The test specimen should be a new component in its original packaging as supplied by the manufacturer.

10.2 *Fittings* — The component must have either 1/4" face seal connections or be of a surface mount design with all metal seals.

10.3 *Sample Size* — The number of samples to be tested is to be determined by the user depending upon their test objectives. It is beyond the scope of this method to determine a statistically valid sample size, however it is recommend that multiple samples of each type are tested.

## 11 Preparation of Apparatus

11.1 Mass Spectrometer-based leak checking of the HCl exposure system after assembly, after the installation of each test component and after opening any connection.

11.2 Before each experiment and during idle times, the apparatus is to be kept in a constant purge condition. The following valve positions would apply during idle times:

VALVE	POSITION
V1	CLOSED
V2	OPEN
V3	OPEN
V4	CLOSED
V5	OPEN
V6	CLOSED
V7	OPEN

11.3 *Moisture Stabilization* — At least four hours prior to the start of a test, a constant flow of wet nitrogen (100 ppm H<sub>2</sub>O) should be established through the sampling port so as to maintain a steady moisture output from the moisture generator. This is accomplished by opening valve V7. During this time, V4 is closed.

11.4 *Moisture Level Verification* — Once the moisture level is stabilized in Section 11.2, the moisture value

measured on the analyzer should be recorded. This level must be 100 ppm  $\pm$  10 ppm or the test cannot proceed.

11.5 Following each test, the section of tubing between V3, V4 and the test piece must be replaced. This will minimize the particle contribution from the system due to system corrosion.

## 12 Calibration

12.1 Calibration of the mass flow controllers should be performed according to manufacturers recommendations. The permeation source used in the moisture generator should be provided with documentation of permeation rate.

## 13 Procedure

13.1 *Baseline Particle Cleanliness Evaluation* — A particle cleanliness evaluation is to be conducted on each component before installation into the HCl exposure system. The baseline particle cleanliness evaluation is to be conducted on new components according to one of the following test methods:

13.1.1 *Low Pressure Regulators* — SEMASPEC #93021510A-STD — Test Method for Determination of Particle Contribution by Low Pressure Regulators in Gas Distribution Systems.

13.1.2 *Valves* — ASTM F1394-92 — Standard Test Method for Determination of Particle Contribution from Gas Distribution System Valves.

13.1.3 *Filters* — SEMASPEC #93021511A — Test Method for Determination of Particle Contribution by Filters in Gas Distribution Systems.

13.2 Install sample in system while maintaining a continuous nitrogen purge of 500 sccm.

13.3 Bake the sample for two hours. Filters will be baked at 100°C and valves and regulators will be baked at 70°C. Maintain a continuous N<sub>2</sub> purge during baking. Valves and regulators will be in the full open position.

13.4 Cool sample overnight under N<sub>2</sub> purge of 500 sccm. From this point on, the sample remains at room temperature.

13.5 Run HCl exposure sequence once per hour six times.

**Table 1 HCl Exposure Sequence**

STEP	TIME	CONDITION
1	5 minutes	Flow dry N <sub>2</sub> , 0.5 slm
2	10 minutes	Flow wet N <sub>2</sub> - 100 ppm H <sub>2</sub> O, 0.5 slm

STEP	TIME	CONDITION
3	5 minutes	Flow dry HCl, 0.5 slm
4	40 minutes	Stagnant HCl, no flow

13.6 Hold sample in static HCl (no flow) for 16 hours.

13.7 Repeat Sections 13.5 and 13.6 for a total of 8 times. This will require a number of days to complete. If this involves a weekend or a period of time when the test must be temporarily suspended, then a dry N<sub>2</sub> flow must be resumed while in this idle mode (see Section 11.1). Flow is not to be interrupted prior to completion of Section 13.6.

13.8 The test should not be interrupted immediately prior to moving to the particle testing, Section 13.9.

13.9 Purge with dry N<sub>2</sub> for 2 hours at 500 sccm.

13.10 Cycle purge for 30 cycles. The cycle purge consists of 20 seconds of rough vacuum 85000 pascals (25" Hg) and 10 seconds of pressurization with dry N<sub>2</sub> to  $5.5 \times 10^5$  pascals (80 psig).

13.11 *Final Dry N<sub>2</sub> Purge* — The component is purged with dry N<sub>2</sub> according to the following schedule: 4 hours for a valve or regulator, and 24 hours for a filter.

13.12 With a purged glove bag around the sample, remove it into the glove bag and cap both ends. The sample can now be removed from the glove bag and transferred to the particle test stand.

13.13 *Final Particle Test in Nitrogen*

13.13.1 Place a purged glove bag on the particle test apparatus at the sample installation point.

13.13.2 Place the sample inside the glove bag and only then remove the caps and install the sample in the system.

13.13.3 Carry out the particle test according to the methods cited in Section 13.1.

## 14 Reporting Results

14.1 *Data Presentation* — Particle data should be plotted for each component on a single graph according to the methods cited in Section 13.1. Both the before HCl exposure and after HCl exposure data should be plotted on the same graph.

14.2 *Other Information* — Other information to be included in the report:

- sample make, model and serial number,
- date of test,
- person who conducted test,



- last calibration date of CNC,
- age of moisture permeation source,
- measured moisture concentration at valve V7,
- ambient temperature and relative humidity, and
- record all exposure times, temperatures, and flow rates from each of the exposure events.

## 15 Related Documents

### 15.1 SEMI Standards

SEMI F55 — Test Method for Determining the Corrosion Resistance of Mass Flow Controllers

### 15.2 SEMATECH Documents<sup>3</sup>

SEMASPEC #90120390B-STD — Test Method for Determination of Particle Contribution by Valves in Gas Distribution Systems.

SEMASPEC #92071233B-STD — “SEMASPEC Provisional Test Method for Determining the Corrosion Resistance of Mass Flow Controllers,” February 5, 1993.

### 15.3 Other

Hwa-Chi Wang, Govind Doddi, and Stephen Chesters, “Comparative Corrosion Studies for HCl - and HBr-Gas Distribution Systems,” 1995 Proceedings - Institute of Environmental Sciences.

Journal of the IES — “Estimating the Lifetime of Electropolished Stainless Steel (EPSS) Tubing in Corrosive Gas Services”, July/Aug. 1994.

The Electrochemical Society, Inc. — “The Role of Moisture in Corrosion of HBr Gas Distribution Systems”, April 1995.

**NOTICE:** SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer’s instructions, product labels, product data sheets, and other relevant literature respecting any materials mentioned herein. These standards are subject to change without notice.

The user’s attention is called to the possibility that compliance with this standard may require use of copyrighted material or of an invention covered by patent rights. By publication of this standard, SEMI takes no position respecting the validity of any patent rights or copyrights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights, are entirely their own responsibility.

---

<sup>3</sup> SEMATECH, 2706 Montopolis Drive, Austin, TX 78741, website: [www.sematech.org](http://www.sematech.org)

# **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](http://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 Section 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 Section 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**

##### *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<sup>1</sup> Standards

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.

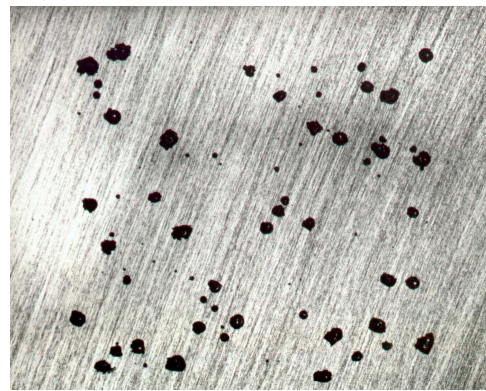
5.1.7 *temperature ramp* — the rate (° C/min) at which the test temperature is increased during the test.

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 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 Section 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.

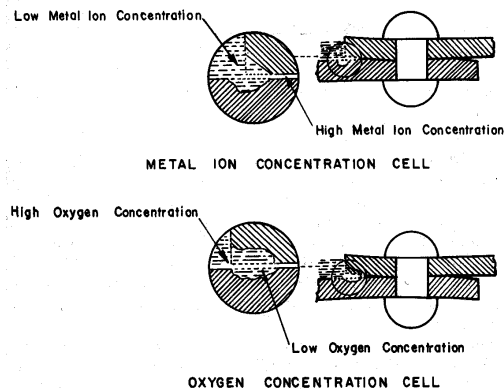


**Figure 1**  
**Examples of Pitting on Type 316 Stainless Steel**  
**After a Pitting Potential Test Performed at 25°C in 1**  
**M NaCl (11 × Magnification)**<sup>(Ref 17.1)</sup>

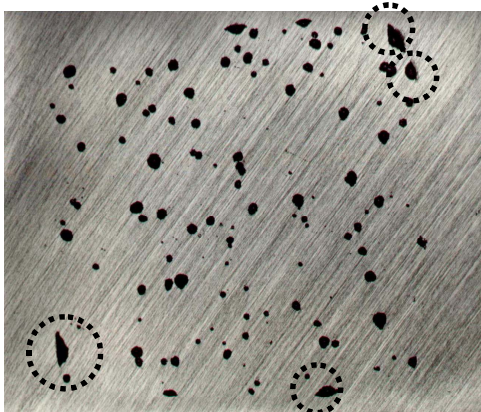
<sup>1</sup> Available from American Society for Testing and Materials, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959. Fax: 1-610-832-9555. Website: <http://www.astm.org>.

## 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 Section 17.2.)



**Figure 3**  
Examples of Crevice Corrosion (Dashed Circles) on  
Type 316 Stainless Steel After a Pitting Potential  
Test in 1 M NaCl at 35°C. 11 × Magnification (See  
Section 17.1.)

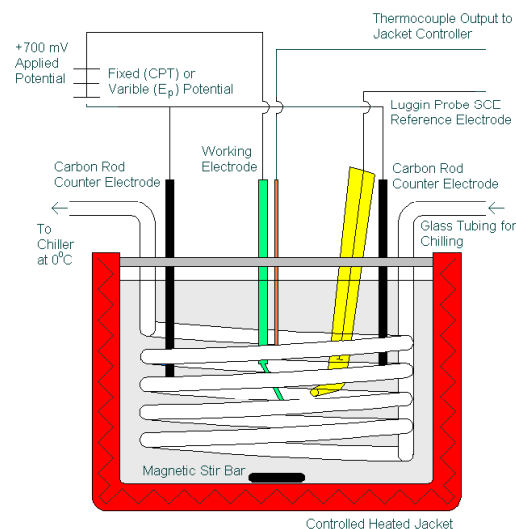
## 8 Apparatus

8.1 In general, refer to ASTM G150 for a detailed description of the apparatus and accessories necessary

to carry out the CPT evaluation. Specifically, Sections 6.1 to 6.9 of ASTM G150 describe the potentiostat, the potential measuring instrument, the current measuring instrument, the temperature controller, and the temperature measurement instrumentation.

8.2 *Flat Specimens* — The test cell for flat specimens (consisting of two options, G5-type cell and a flushed-port cell), are given in Appendices X2 and X3 of ASTM G150 with a detailed description of each.

8.3 *Tubular Specimens* — The test cell can be modified for testing tubular specimens. The modified test cell consists of a 2-L glass beaker cut to 4.5 in. high and capped with a polyethylene cover. A three-electrode set up is used consisting of the test specimen, an SCE reference electrode inserted in a Luggin probe, and two graphite rods as counter electrodes. See Figure 4 for the modified test cell set-up.



**Figure 4**  
Modified Test Cell for CPT Testing of Tubular  
Specimens; Tubing Specimen is the Working  
Electrode

## 9 Test Specimens

9.1 *Flat Specimens* — In general, refer to ASTM G150 for a detailed description of the flat specimen.

9.2 *Tubing Specimens* — Tubing specimens consist of 2.0-in. lengths of tubing. The tubing should be faced, and the cut ends should be masked with a non-reactive lacquer.

9.3 All specimens should be masked to isolate the test to the surface of interest only. This can be accomplished by masking with a non-reactive lacquer, which can be baked to ensure optimum adhesion.

Alternately, plater's tape can also be used, leaving 0.25 in. uncovered at the end for electrical contact.

**9.4 Finish** — In general, refer to ASTM G150 for details on the typical finish of the test specimens. Since surface finish can effect the CPT results, surface finished should be done as consistently as possible within a sample set. Details of the surface finishing process, parameters and characterization should be provided in the test report as outlined in Section 15.

**9.5 Sampling** — It is recommended that a minimum of 3 specimens, and preferably 5 or more, should be used to obtain statistical significance in the testing results.

**9.6 Test Area** — In general, refer to ASTM G150, which states a minimum test area of 1 cm<sup>2</sup>. This minimum test area is required for both flat specimens and tubing specimens.

## 10 Reagents And Materials

**10.1** In general, refer to Section 8 of ASTM G150 describing the purity of the reagents and water needed for the test solution as well as the purity of the N<sub>2</sub> purging gas.

**10.2 Standard Test Solution** — Comparison of the CPT results requires consistency in the test solution, so it is recommended that a 1 M (mole/liter) sodium chloride (NaCl) electrolyte be used as electrolyte. However, because of the different processes gases used by the semiconductor industry (HCl, HBr, Cl<sub>2</sub>, BCl<sub>3</sub>, HF, WF<sub>6</sub>), it is possible to substitute another test solution to represent the particular anion of the process gas which may impart different pitting characteristics (e.g., 1 M NaBr, or 1M NaF).

## 11 Applied Potential

**11.1 Stainless Steel** — In general, refer to section 9 of ASTM G150, which provides the standard anodic potential commonly used for most stainless steels (an anodic potential of 700 mV versus SCE at 25°C). Section 9 also provides an alternative potential if there is uncertainty as to whether this potential is sufficiently high to obtain a potential independent CPT.

**11.2 Alternative Alloys** — Alloys other than 316L stainless steel can be tested using the applied potential given in Section 11.1.

## 12 Procedure

**12.1** In general refer to Section 10 of ASTM G150 for details on specimen mounting, cleaning, and placement,

test solution preparation, test procedure, and completion of the test.

**12.2** Refer to Annexes A1 and A2 in ASTM G150 for guidelines to calibrate the specimen temperature.

**12.3 Tubing specimens** — Tubing testing requires different specimen mounting and preparation than given in Section 9 of ASTM G150. Tubing specimens should be placed vertically in solution so that approximately one inch is submerged.

## 13 Visual Examination Of Test Specimen

**13.1** In general, refer to Section 11 of ASTM G150 for a description of the visual examination of the test specimen.

## 14 Data Analysis

**14.1** In general, refer to Section 12 of ASTM G150 for details on evaluating the critical pitting temperature.

## 15 Reporting Results

**15.1** Section 13 of ASTM G150 provides a list of mandatory information required in the test report.

**15.2** Other mandatory information that should be provided are the starting test electrolyte composition, the diameter of the tubing for tubing specimens, identification of the surface finish process (e.g., electropolish, passivation), and any other specific parameters or characterization related to the surface finish process. Welded and non-welded specimens should also be clearly identified, as well the welding procedure used to weld the specimen.

## 16 Precision and Bias

**16.1** The precision of this test method is yet to be determined

**16.2** The bias of this test method is yet to be determined.

## 17 References

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

**17.2** Philip A. Schweitzer, Ed. "Corrosion and Corrosion Protection Handbook." Marcel Dekker, New York, p. 11, 1983.

## APPENDIX 1

### EXAMPLE OF A CPT TEST PROCEDURE

**NOTICE:** The material in this appendix is an official part of SEMI F77 and was approved by full letter ballot procedures.

#### A1-1 Principle

A1-1.1 Critical pitting temperature (CPT) is an electrochemical test that determines the localized corrosion resistance of stainless steel and other alloys. The test is performed in a solution of sodium chloride (NaCl). The sample is polarized at 700 mV<sub>SCE</sub> while the temperature is gradually increased from 0°C at a rate of 1°C/min. Above some critical temperature, pitting corrosion will occur signified by a rapid increase in measured current above 100  $\mu$ A/cm<sup>2</sup> for 60 seconds. The experiment is monitored and controlled using special software.

#### A1-2 Precautions

A1-2.1 Use caution with heating mantles, as they can become very hot.

#### A1-3 Equipment and Supplies

A1-3.1 Corrosion Test System (potentiostat and software)

A1-3.2 Chiller (capable of -10°C to 100° C)

A1-3.3 Temperature controller

A1-3.4 Heating Mantle

A1-3.5 Stir Plate/Bar (Immersible)

A1-3.6 Glass cell (adapted to contain electrodes, probes, and cooling coil)

A1-3.7 Counter electrodes (graphite rods)

A1-3.8 Reference electrode (saturated calomel) and luggin probe

A1-3.9 Cooling coils

#### A1-4 Chemicals and Reagents

A1-4.1 Sodium Chloride (ACS Reagent Grade)

A1-4.2 18 M $\Omega$  deionized water

#### A1-5 Procedure

A1-5.1 Turn chiller on and set at -2°C.

A1-5.2 Fill Beaker with 800 ml of 1 M (mole/liter) NaCl electrolyte (Preferably chilled in refrigerator). Turn stir plate on and ensure adequate stirring with stir bar.

A1-5.3 Turn on temperature controllers. Adjust set point to -1°C. Place lid on beaker and ensure temperature probe is in solution.

A1-5.4 Place two counter electrodes and one reference electrode in solution. The reference electrode should be placed in a luggin probe. The luggin probe tip should be placed as close to the sample as possible without interfering with the stir bar. Connect electrical wires to counter and reference electrodes.

A1-5.5 Turn on computer and potentiostat and start software.

A1-5.6 After temperature has dropped to 0°C, insert specimen into test cell. One inch of the sample for tube stubs should be submerged. Temperature probe should be touching sample (masked section only).

A1-5.7 Connect electrical wires to specimen and start the test.

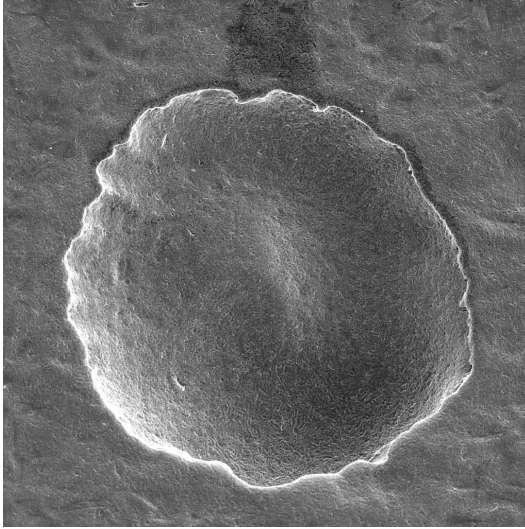
A1-5.8 After approximately 500 seconds during the initial delay, decrease stir bar rotation speed by one half.

A1-5.9 After temperature has increased to 10°C, periodically monitor temperature to ensure proper heating rate.

A1-5.10 When test is completed, remove the specimen and rinse with DI water.

A1-5.11 Examine specimen for pit location. If pit is touching the masked-off or lacquered area, the test is not valid. See Figure A-1.1 for a SEM micrograph of a typical pit from a valid CPT test.

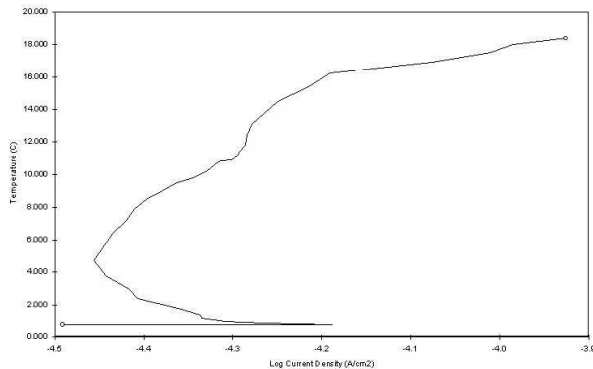
A1-5.12 Determine critical pitting temperature. The critical pitting temperature is the temperature at which the current density exceeds 100  $\mu$ A/cm<sup>2</sup> for at least 60 seconds. Figure A-1.2 shows an example of a temperature vs. current density scan. In this particular example, the critical pitting temperature was determined to be 18°C.



**Figure A-1.1**  
SEM Micrograph of Pit Formed During CPT Testing. The Pit is Approximately 100 Microns Across.

**NOTICE:** SEMI makes no warranties or representations as to the suitability of the standards set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

By publication of this standard, Semiconductor Equipment and Materials International (SEMI) takes no position respecting the validity of any patent rights or copyrights asserted in connection with any items mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights are entirely their own responsibility.



**Figure A-1.2**  
Current Density as a Function of Temperature. The Critical Pitting Temperature is the Temperature at Which the Current Density Exceeds  $100 \mu\text{A}/\text{cm}^2$  for At Least 60 Seconds.



# SEMI F78-0304

## PRACTICE FOR GAS TUNGSTEN ARC (GTA) WELDING OF FLUID DISTRIBUTION SYSTEMS IN SEMICONDUCTOR MANUFACTURING APPLICATIONS

This practice 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 October 16, 2003. Initially available at [www.semi.org](http://www.semi.org) February 2004; to be published March 2004. Originally published July 2003.

### 1 Purpose

1.1 The purpose of this practice is to provide procedures for welding stainless steels and other corrosion resistant metals and alloys (CRAs) for fluid (liquid or gas) distribution systems in semiconductor manufacturing applications. Welds performed following these procedures are of sufficient quality to provide the required system purity, weld integrity, and weld strength for use in semiconductor manufacturing applications.

### 2 Scope

2.1 This practice provides procedures for gas tungsten arc (GTA) autogenous butt joint welds of stainless steel and other CRAs in fluid distribution systems. The fluid distribution system includes tubing, pipe, fittings, valves, subassemblies and components that contain and distribute fluid.

**NOTICE:** This practice 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 The stainless steels covered by this practice are limited to the austenitic and superaustenitic grades of stainless steel.

3.2 Corrosion resistant metals and alloys covered by this practice are limited to solid solution grades of nickel alloys and solid solution grades of titanium alloys.

3.3 This practice applies only to autogenous GTA circumferential butt joint welds performed on fluid distribution system components 6 inches or less in diameter.

3.4 This practice applies only to automatic, mechanized, or machine GTA welding processes.

3.5 This practice applies only to welds performed with no fillers and no fluxes.

3.6 This practice does not apply to pressure vessel or process chamber welds.

### 4 Referenced Standards

NOTE 1: The following documents become part of the practice to the extent that they are included herein.

#### 4.1 SEMI Standards

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

SEMI F81 — Specification for Visual Inspection and Acceptance of Gas Tungsten Arc (GTA) Welds in Fluid Distribution Systems in Semiconductor Applications

#### 4.2 ANSI/ASME Standards<sup>1</sup>

ANSI/ASC Z49.1 — Safety in Welding, Cutting, and Allied Processes

BPE — Bioprocessing Equipment Standard

B16.25 — Butt Welding Ends

B31.3 — Process Piping

Boiler and Pressure Vessel Code — Section IX, Qualification Standard for Welding and Brazing Procedures, Welders, Brazers, and Welding and Brazing Operators

#### 4.3 ASTM Standards<sup>2</sup>

A269 — Specification for Seamless and Welded Austenitic Stainless Steel Tubing for General Service

---

1 American National Standards Institute, New York Office: 11 West 42nd Street, New York, NY 10036, USA. Telephone: 212.642.4900; Fax: 212.398.0023 Website: [www.ansi.org](http://www.ansi.org)

2 American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428-2959, USA. Telephone: 610.832.9585, Fax: 610.832.9555, Website: [www.astm.org](http://www.astm.org)



A450 (Section 25) — General Requirements for Carbon, Ferritic Alloy, and Austenitic Steel Tubes

A632 — Specification for Seamless and Welded Austenitic Stainless Steel Tubing (Small Diameter) for General Service

#### 4.4 AWS Standards<sup>3</sup>

AWS A3.0 — Standard Welding Terms and Definitions

AWS B2.1 — Specification and Qualification of Welding Procedures and Welders for Piping and Tubing

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

## 5 Terminology<sup>4</sup>

### 5.1 Definitions

5.1.1 *angular misalignment* — the condition that exists when the tubing angle is unintentionally changed at the weld.

5.1.2 *autogenous weld* (2) — a fusion weld made without filler metal.

5.1.3 *automatic arc welding downslope* — the time during which the welding current is reduced continuously from the final level until the arc is extinguished.

5.1.4 *axial misalignment* — the offset caused by tubing being in line but not centered at the weld.

5.1.5 *backing gas* — an inert gas (or gas mixture) on the interior of the weld joint used to prevent or reduce formation of oxides and other detrimental surface substances during welding, and to provide pressure for weld profile.

5.1.6 *bead* (2) — non-standard term for *weld bead*.

5.1.7 *bead overlap* — in a pulsed weld the amount of coverage of a weld pulse of the previous weld pulse, usually measured in percentage of the diameter of the pulse.

5.1.8 *bead variation* — the amount of change of ID bead width from one area to another.

5.1.9 *bead width* — the width of the weld bead on the ID, normally measured in units of  $T$ , where  $T$  is the nominal tube wall thickness.

5.1.10 *center line shrinkage* — a profile-reducing defect or discontinuity normally formed by shrinkage during solidification.

5.1.11 *color* — the darkness of the oxidation of the weld or adjacent surfaces. Non-standard term for *discoloration*.

5.1.12 *color line* — acceptance criteria of the maximum amount of discoloration allowed on the weld or adjacent surfaces.

5.1.13 *concavity* (3) — a condition in which the surface of a weld is depressed relative to the surface of the tube or pipe. Concavity is measured as a maximum distance from the outside or inside diameter surface of a weld along a line perpendicular to a line joining the weld toes.

5.1.14 *convexity* (3) — a condition in which the surface of a weld is extended relative to the surface of the tube or pipe. Convexity is measured as a maximum distance from the outside or inside diameter surface of a weld along a line perpendicular to a line joining the weld toes.

5.1.15 *coupon* — weld sample which is opened for inspection to insure that the weld meets specifications.

5.1.16 *coupon-in* — first coupon prior to production welding of butt weld joint.

5.1.17 *coupon-out* — last coupon after production welding of butt weld joint ends.

5.1.18 *discoloration* (3) — any change in surface color from that of the base metal. Usually associated with *oxidation* occurring on the weld and heat affected zone on the outside and inside diameter of the weld joint as a result of heating the metal during welding. Colors may range from pale bluish-gray to deep blue, and from pale straw color to a black crusty coating.

5.1.19 *downslope* — See *automatic arc welding downslope*.

5.1.20 *dross* (2) — non-standard term for *slag*.

5.1.21 *electrode* (2) — non-standard term for *tungsten electrode*.

5.1.22 *enclosed weld head* — weld head in which the weld joint is held and welded within a closed chamber containing a shielding purge gas.

---

3 American Welding Society, 550 NW LeJeune Road, P.O. Box 351040, Miami, Florida 33135, USA. Telephone: 800.443.9353, Website: [www.aws.org](http://www.aws.org)

4 The terminology has been derived from the following sources:

- (1) Webster's New World College Dictionary Fourth Edition
- (2) ANSI/AWS A3.0 Standard Welding Terms and Definitions
- (3) ASME BPE Bioprocessing Equipment Standard

5.1.23 *encroachment* — non-standard term for ID convexity.

5.1.24 *examiner* — a person who performs examination of a particular object, or evaluates an operation, for compliance to a given standard. The examiner performs quality control for the manufacturer, fabricator, or erector.

5.1.25 *fluid* (1) — liquid or gas.

5.1.26 *gas* (1) — the fluid form of a substance in which it can expand indefinitely and completely fill its container; form that is neither liquid or solid.

5.1.27 *gas tungsten arc welding (GTAW)* (3) — an arc welding process that uses an arc between a tungsten electrode (nonconsumable) and the weld pool. The process is used with a shielding gas.

5.1.28 *halo* — non-standard term for *discoloration* resulting from welding procedure.

5.1.29 *haze* — non-standard term for *discoloration* resulting from welding procedure.

5.1.30 *heat-affected zone (HAZ)* (2) — the portion of the base metal whose mechanical properties or microstructure have been altered by the heat of welding.

5.1.31 *heat tint/color* — non-standard term for *discoloration* resulting from welding procedure.

5.1.32 *inclusion* (2) — entrapped foreign solid material, such as slag, flux, tungsten, or oxide.

5.1.33 *inert gas* — a gas that normally does not combine chemically with materials. A protective atmosphere.

5.1.34 *inspector* — a person who verifies that all required examinations and testing have been completed, and who inspects the assembly to the extent necessary to be satisfied that it conforms to all applicable examination requirements. The inspector performs quality assurance for the owner. The inspector is designated by the owner and shall be the owner, an employee of the owner, an employee of an engineering or scientific organization, or of a recognized insurance or inspection company acting as the owner's agent.

5.1.35 *lathe welding* — automatic or machine welding of tubes or pipes in which the electrode is stationary and the weld joint rotates. Lathe welding as defined here is a fusion process without the addition of filler.

5.1.36 *liquid* (1) — having its molecules moving freely with respect to each other so as to flow readily, unlike a solid, but because of cohesive forces not expanding infinitely like a gas.

5.1.37 *liquid cylinder* — often referred to as a dewar, an insulated and pressure controlled metal cylinder used to store fluids in their liquid form.

5.1.38 *meandering* (3) — of or pertaining to a weld bead that deviates from side to side across the weld joint rather than tracking the joint precisely.

5.1.39 *orbital welding* (3) — automatic or machine welding of tubes or pipes in-place with the electrode rotating (or orbiting) around the work. Orbital welding, as it applies to this standard, is a fusion process without the addition of filler.

5.1.40 *oxidation* (3) — the formation of an oxide layer on a metal surface. When excessive oxidation occurs as a result of welding, it is visible as *discoloration*.

5.1.41 *oxide island* — non-standard term for *slag*.

5.1.42 *pressure cylinder* — a metal cylinder used to store gases under pressure.

5.1.43 *profile defect* — any defect or discontinuity that reduces the wall thickness below that of the parent metal.

5.1.44 *pulsed gas tungsten arc welding* — a gas tungsten arc welding process variation in which the current is varied in regular intervals.

5.1.45 *purge* — the application of an inert gas (or gas mixture) to the OD or ID surface of the weld joint to displace non-inert atmospheric gases. A block purge is a non-flowing purge with positive pressure.

5.1.46 *purge gas* — an inert gas (or gas mixture) used to displace the ambient atmosphere from the inside (ID) of the weld joint.

5.1.47 *root* — non-standard term for *root surface*.

5.1.48 *root surface* (2) — the exposed surface of a weld opposite the side from which the welding was done.

5.1.49 *rotation delay* — time delay between when the arc is initiated and the rotor begins to turn.

5.1.50 *shield gas* — inert gas (or gas mixture) that protects the electrode and molten puddle from atmosphere and provides the required arc characteristics.

5.1.51 *slag* (2) — a non-metallic product resulting from the mutual dissolution of non-metallic impurities in some welding processes.

5.1.52 *tack weld* (2) — a weld made to hold the parts of a weldment in proper alignment until the final welds are made.

5.1.53 *tail-out* (2) — non-standard term for *automatic arc welding downslope*.