

APPENDIX 1

NOTE: The material in this appendix is an official part of SEMI F64 and was approved by full letter ballot procedures on April 30, 2001 by the Facilities Regional Standards Committee.

A1-1 Data Analysis: Method A-1

Table A1-1 Sample Data Sheet: Methods A-1 and A-2

Date: _____ Time: _____
D.U.T. # _____ Ambient Temp _____ °C
Standard Flow Device: _____
Full Scale: _____ sccm
Ramp Rate (Method A-2) _____ kPa/sec

<i>Time (sec)</i>	<i>P_I (psig)</i>	<i>Q_A (sccm)</i>	<i>Q_I (sccm)</i>
0	25.01	50.00	50.01
0.05	25.01	50.01	50.01
0.10	25.02	50.00	50.02
0.15	25.01	49.99	50.01
.	.	.	.
.	.	.	.
.	.	.	.
2.20	25.40	52.68	51.38
2.25	25.85	55.23	52.56
.	.	.	.
.	.	.	.
.	.	.	.
6.30	26.89	46.28	49.23

A1-1.1 Data Interpretation, Method A-1

A1-1.1.1 δQ_+ = maximum positive deviation of actual flow from nominal

$$\delta Q_+ = Q_{max} - Q_N$$

where:

Q_{max} = maximum value of Q_A

Q_N = average value of Q_A during time when P_I = initial steady state

δQ_- = maximum negative deviation of actual flow from nominal

$$\delta Q_- = Q_{min} - Q_N$$

where:

Q_{min} = minimum value of Q_A

For example (see Table A1-1), if $Q_N = 50.01$ sccm, $Q_{max} = 55.23$ sccm, and $Q_{min} = 46.28$ sccm, then

$$\delta Q_+ = 55.23 \text{ sccm} - 50.01 \text{ sccm} = 5.22 \text{ sccm}$$

$$\delta Q_- = 46.28 \text{ sccm} - 50.01 \text{ sccm} = -3.73 \text{ sccm}$$

A1-1.1.2 t_s is settling time to Q_N ; i.e.,

$$t_s = \text{elapsed time from initiation of pressure transient } (t_o) \text{ to when } Q_A \text{ is within 0.5\% of reading of } Q_N \text{ } (t_f) = t_f - t_o$$

δM is deviation of mass of material delivered during the disturbance relative to steady state mass delivery; i.e.,

$$\delta M = \frac{\int_{t_i=t_o}^{t_f} (Q_A - Q_N)(t_{ih} - t_i) dt}{60 \text{ sec / min}} \quad (2)$$

This quantity may be thought of as the net area between the Q_A versus time curve and the Q_N versus time line. See Figure A1-1 below.

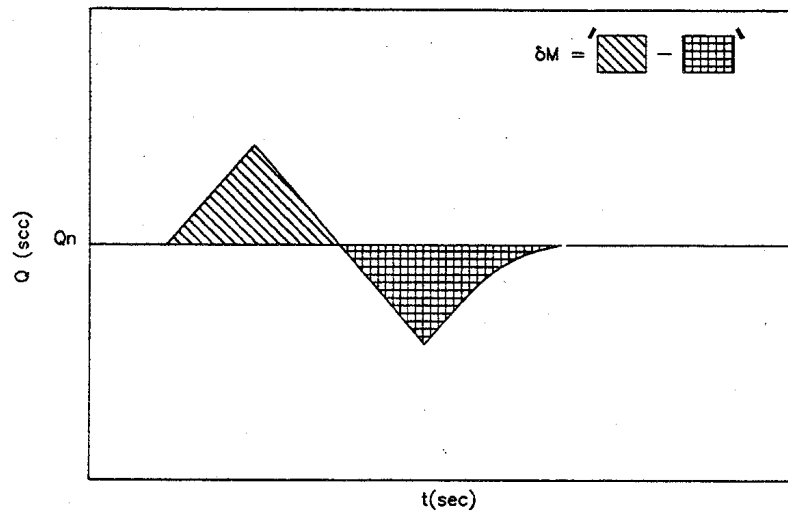


Figure A1-1
 Q_N versus t (sec)

NOTE 1: δM is expressed in units of sccm.

A1-2 Data Analysis: Method A-2

Refer again to Table A1-1

A1-2.1 Data Interpretation, Method A-2

Q_R is the steady state actual flow while the inlet pressure is being ramped. It is calculated by averaging Q_A values from time $t_o + 50$ sec to $t_o + 110$ sec.

ΔQ is the steady state deviation of actual flow during the inlet pressure ramp from that while inlet pressure is constant; i.e.,

$$\Delta Q = Q_R - Q_N \quad (3)$$



V_{eq} is the equivalent internal control volume of the DUT. Note that it may not represent the actual internal volume of the DUT; i.e.,

$$V_{eq} = \frac{\Delta Q}{P_1} \times \left(\frac{14.7 \text{ psi/std. atm}}{60 \text{ sec/min}} \right) \quad (4)$$

For example, if $\Delta Q = 5.02 \text{ sccm}$ and $P_1 = 0.7 \text{ kPa (0.1 psi)/sec}$, then

$$V_{eq} = [(5.02 \text{ sccm}) / (0.7 \text{ kPa (0.1 psi)/sec})] \times [(101.325 \text{ kPa (14.7 psi)/std. atm}) / (60 \text{ sec/min})] = 12.3 \text{ ccm}$$

A1-3 Data Analysis: Method B

Table A1-2 Sample Data Sheet: Method B

Date: _____ Time: _____
DUT # _____ Ambient Temp _____ °C
Standard Flow Device _____
Full Scale _____ sccm

P_I (psig)	Setpoint Flow (sccm)	Q_A (sccm)	Q_I (sccm)
15.02	0	0.00	-0.01
15.00	50	50.01	50.01
15.01	100	100.12	100.01
50.06	0	0.00	-0.05
50.04	50	50.12	50.01
50.03	100	100.56	100.02

A1-3.1 Data Interpretation, Method B

PC_o is the pressure coefficient of indicated flow per pressure change at zero flow; i.e.,

$$PC_A (\%) = \frac{\Delta Q_A}{\Delta P_I Q_{sp}} \times 100 \quad (5)$$

$$PC_o (\%) = \frac{Q_{If} - Q_{li}}{(P_{If} - P_{li}) Q_{FS}} \times 100 \quad (6)$$

PC_A is the pressure coefficient of actual flow per pressure change at a setpoint; i.e.,

For example, from Table A2.1,

$$PC_o (\%) = \{[-0.05 \text{ sccm} - (-0.01 \text{ sccm})] / [(50.06 \text{ psig} - 15.02 \text{ psig})(100 \text{ sccm})]\} \times 100$$

$$= -0.0011\% \text{ of FS/psi}$$

$$PC_A (@ 50\%) = [50.12 \text{ sccm} - 50.01 \text{ sccm}] / [(50.04 \text{ psig} - 15.00 \text{ psig})(50 \text{ sccm})] \times 100$$

$$= 0.0063\% \text{ of reading/psi at 50 sccm}$$

$$PC_A (@ \text{FS}) = [100.56 \text{ sccm} - 100.12 \text{ sccm}] / [(50.03 \text{ psig} - 15.01 \text{ psig})(100 \text{ sccm})] \times 100$$

$$= 0.013\% \text{ of reading/psi at 100 sccm}$$



A-4 Data Analysis: Method C

Table A4-1 Sample Data Sheet: Method C

Date: _____ Time: _____
DUT # _____ Ambient Temp _____ °C
Standard Flow Device _____
Setpoint _____ sccm
Te = _____ °C P1 = _____ psig

<i>Time (sec)</i>	<i>P2 (kPa)</i>	<i>Q_A (sccm)</i>	<i>Q_I (sccm)</i>
0	101.338	50.05	50.0
0.05	101.338	50.05	50.0
.	.	.	.
.	.	.	.
.	.	.	.
2.20	95.086	52.28	51.5
2.25	88.406	54.56	52.1
.	.	.	.
.	.	.	.
.	.	.	.
8.45	32.424	50.08	50.0
8.50	31.811	50.07	50.0
.	.	.	.
.	.	.	.
.	.	.	.
15.20	26.838	50.06	50.0

A1-4.1 Data Interpretation, Method C – See Appendix A1-1.1

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SEMI F65-1101

DIMENSIONAL SPECIFICATION FOR MOUNTING BASES OF DIAPHRAGM VALVES USED WITH METRIC PFA TUBES

This specification was technically approved by the Global Liquid Chemical Distribution Systems Committee and is the direct responsibility of the Japanese Liquid Chemical Distribution Systems Committee. Current edition approved by the Japanese Regional Standards Committee on August 3, 2001. Initially available at www.semi.org September 2001; to be published November 2001.

1 Purpose

1.1 This document specifies dimensions of mounting base and their clearance hole sizes for bolts and the hole locations of diaphragm valves used with metric PFA tubes in liquid chemical distribution facilities and process equipment for semiconductor and flat panel display manufacturing.

1.2 To avoid any disturbance of future development and to facilitate interchangeability of diaphragm valves, this document has a limited scope as specified in Section 2.

2 Scope

2.1 This document applies to mounting bases for two way valves with a diaphragm designed to shut off and/or regulate a liquid chemical flow in tubes whose sizes are listed in Table 1.

2.2 The valves are made from materials such as PTFE or PFA, which have high corrosion resistance and low contamination contribution to the fluid.

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

3 Referenced Standards

3.1 ISO Standards¹

ISO 273 — Fasteners — Clearance Holes for Bolts and Screws

ISO 2768-1 — General Tolerance — Part 1: Tolerances for linear and angular dimensions without individual tolerance indications

3.2 JIS Standards²

JIS B 1001 — Diameter of Clearance Holes and Counterbores for Bolts and Screws

JIS B 0405 — General Tolerance — Part 1: Tolerances for linear and angular dimensions without individual tolerance indications

NOTE 1: Unless otherwise indicated, all documents cited shall be the latest published versions.

4 Terminology

4.1 Abbreviations and Acronyms

4.1.1 PFA — tetrafluoroethylene perfluoroalkylvinylether copolymer

4.1.2 PTFE — polytetrafluoroethylene

4.2 Definitions

4.2.1 back pressure — a maximum allowable pressure applied to outlet of a diaphragm valve.

4.2.2 liquid chemicals — acid, alkali, organic solvent, and pure water used for wet stations; resists and developers used for track system; and other chemicals used for process or maintenance (such as slurry of chemical-mechanical polishing) of equipment or facilities.

4.2.3 mounting bases — plates which are attached to diaphragm valves to mount the valves to equipment or a facility.

5 Dimensional Specification

5.1 Mounting Base Dimensions — the length (l) and width (w) of the mount base plate indicated in the Figure 1 and the values for different tube sizes are specified in Table 1.

5.2 Clearance Hole Locations — the location of the clearance holes for bolts are indicated with “w” and “l” in the Figure 1 and the values for different tube sizes are specified in Table 1.

5.3 Clearance Hole Sizes — the size of clearance holes (ϕD) for different tube sizes are specified in the Table 1 according to ISO 273 (JIS B 1001).

¹ International Organization for Standardization (ISO), 1, rue de Varembe, Case postale 56, CH-1211 Genève 20, Switzerland.

² Japan Standards Association, 4-1-24, Akasaka, Minato-ku, Tokyo, Japan 107-8440.

5.4 Where the dimension depends on the valve design considering the back pressure, the upper row in Table 1 applies to the valves that withstand up to 0.2 megapascals (MPa) (29 pounds per square inch (psi)) back pressure, and the lower row applies to the valves that withstand more than 0.2 MPa (29 psi) back pressure.

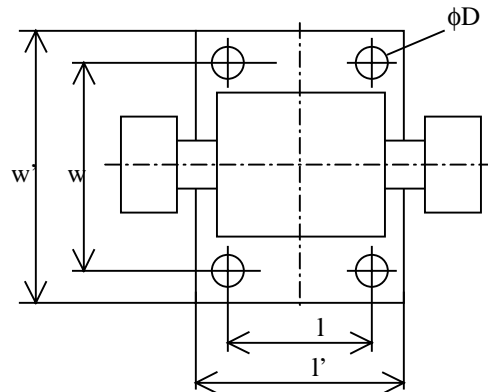


Figure 1
Top View of A Typical Diaphragm Valve with A Mounting Base

Table 1 Dimensional Specifications

Tube Size (mm) (OD / ID)*	Back Pressure (Mpa)	l' (mm)	l (mm)	w' (mm)	w (mm)	ϕD (mm)	Bolt Type (example)
25 / 22	≤ 0.2	60	48	96	84	7	M6
	> 0.2	68	56				
19 / 16	≤ 0.2	44	32	76	64	7	M6
	> 0.2	52	40	84	72		
12 / 10	≤ 0.2	34	22	62	50	7	M6
	> 0.2	46	34	70	58		
10 / 8	≤ 0.2	34	22	62	50	7	M6
	> 0.2	46	34	68	56		
6 / 4	≤ 0.2	32	20	56	44	7	M6
	> 0.2	40	28	60	48		

Note 1: OD and ID are outside diameter and inside diameter of the tube respectively.

Note 2: Refer to ISO 2768-1 or JIS B 0405 for tolerances.

6 Measurements

6.1 The mounting base must be conditioned for a minimum of one hour in an air environment of $23 \pm 3^{\circ}\text{C}$ ($73.4 \pm 5.4^{\circ}\text{F}$) prior to measuring the dimensions.

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SEMI F66-1101

SPECIFICATION FOR PORT MARKING AND SYMBOL OF STAINLESS STEEL VESSELS FOR LIQUID CHEMICALS

This specification was technically approved by the Global Liquid Chemical Distribution Systems Committee and is the direct responsibility of the Japanese Liquid Chemical Distribution Systems Committee. Current edition approved by the Japanese Regional Standards Committee on August 3, 2001. Initially available at www.semi.org September 2001; to be published November 2001.

1 Purpose

1.1 This document specifies port marking and symbol of stainless steel vessels for liquid chemicals used in semiconductor and flat panel display manufacturing equipment and liquid chemical distribution facilities.

2 Scope

2.1 This document covers stainless steel vessels with tubes that penetrate into the vessels as inlets or outlets of liquid chemical to/from the vessels.

2.2 A vessel which uses coupling (quick coupling) at its tube end (port) is excepted from the scope.

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

3 Referenced Standards

3.1 None.

4 Terminology

4.1 *dip tube* — a piece of tube which penetrates into a vessel as an inlet or outlet of liquid chemical to/from the vessel.

4.2 *liquid chemical* — organic or inorganic liquid chemical used for semiconductor or flat panel display manufacturing.

4.3 *port* — an end of a tube attached to a vessel.

5 Requirements for Port Marking

5.1 The dip tube port shall be marked as “DIP”. If more than one dip tube port exists on a vessel, each port shall be marked as “DIP”.

5.2 *Additional Marking* — A description to identify multiple ports may be added to the dip port marking such as DIP-1 or DIP (1).

5.3 *Location of Marking* — “DIP” marking shall be located where it will clearly identify the dip tube port. If more than one dip tube ports exist on a vessel, each

marking shall be located so that the corresponding port is clearly identified.

5.4 *Method of Marking* — The marking shall be engraved, etched, or labeled properly so that the “DIP” marking shall not be removed. Color of the marking is optional.

5.5 *Size of Marking* — The marking shall be sized so that it is clearly readable.

6 Requirements for Symbol

6.1 A symbol illustrated in Figure 1 shall be used to draw a stainless steel vessel with a dip tube. A dip tube shall be identified in the symbol as a line penetrating into a rectangle.

6.2 If more than one dip tube exists on a vessel, the symbol shall have the same number of lines which correspond to the dip tubes.

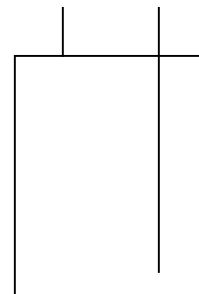


Figure 1
Symbol for Stainless Steel Vessel with a Dip Tube

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SEMI F67-1101

TEST METHOD FOR DETERMINING INERT GAS PURIFIER CAPACITY

This test method 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 27, 2001. Initially available at www.semi.org September 2001; to be published November 2001.

1 Purpose

1.1 The purpose of this document is to define a test method to quantify impurity removal capacity of inert gas purifiers.

2 Scope

2.1 To determine the impurity capacity of a gas purifier at the point of breakthrough. Capacity tests are done by adding ppm levels of a given gaseous impurity to a pure zero gas and monitoring the effluent of the test purifier for active impurity species.

NOTE 1: Mixtures of two or more impurities for multi impurity removal purifiers is a more representative method for determining capacity.

2.2 This document is intended for point of use (POU) inert gas purifiers where inlet purity is 99.9995% or higher.

2.3 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 and safety health practices and determine the applicability of regulatory limitations prior to use.

3 Limitations

3.1 The inherent limitation to this method is the limit of detection (LOD) of the analytical instrument employed by the user.

3.2 This test method can only be used to compare purifier capacity results if the user application for flow rate, pressure, and temperature are the same as the test conditions. Different users and/or different operating conditions may result in different purifier performance results.

3.3 In testing mixtures of impurities, some impurities may influence the capacity results. Discussion with the manufacturer is highly recommended prior to testing.

3.4 The test method does not apply to particulates.

3.5 This test method can only be used to compare the capacity of different purifiers, when the purifiers are sized for the appropriate flow rate. Comparing purifiers of different maximum flow ratings will result in misleading information.

3.6 This test method will provide capacity information only for impurities that are used in the challenge gas.

4 Referenced Standards

4.1 SEMI Standards

SEMI E29 — Standard Terminology for the Calibration of Mass Flow Controllers and Mass Flow Meters

SEMI F6 — Guide for Secondary Containment of Hazardous Gas Piping Systems

SEMI F22 — Guide for Gas Distribution Systems

SEMI F33 — Method for Calibration of Atmospheric Pressure Ionization Mass Spectrometer (APIMS)

4.2 ANSI Standards¹

ANSI B46.1 — Surface Texture (Surface Roughness, Waviness, and Lay)

NOTE 2: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 Abbreviations and Acronyms

5.1.1 APIMS — atmospheric pressure ionization mass spectrometer

5.1.2 °C — degrees Celsius

5.1.3 DUT — device under test

5.1.4 °F — degrees Fahrenheit

5.1.5 in — inch

5.1.6 kPa — kiloPascal

5.1.7 LOD — limit of detection

5.1.8 m — meter

5.1.9 MFC — mass flow controller

5.1.10 NMHC — non methane hydrocarbons

5.1.11 POU — point of use

5.1.12 ppb — parts per billion, volume basis

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

5.1.13 *ppm* — parts per million, volume basis

5.1.14 *psi* — pounds per square inch

5.1.15 *psia* — pounds per square inch absolute

5.1.16 *psig* — pounds per square inch gauge

5.1.17 R_a — surface roughness average (as defined in ANSI B46.1)

5.1.18 $R_{a,max}$ — surface roughness maximum (as defined in ANSI B46.1)

5.1.19 *s* — second

5.1.20 *sccm* — standard cubic centimeters per minute

5.1.21 *slpm* — standard liter per minute

5.2 Definitions

5.2.1 *activation* — the process of initially preparing the purifier media to be chemically reactive with gas impurities.

5.2.2 *activation temperature* — temperature at which DUT was initially prepared.

5.2.3 *atmospheric pressure ionization mass spectrometer (APIMS)* — an instrument consisting of an atmospheric pressure ion source where gas phase impurities are ionized via charge exchange reactions with the bulk gas. These ions are directed into a vacuum chamber where they are then separated by a mass analyzer and detected by an electron multiplier.

5.2.3.1 *ion source* — the section of a mass spectrometer used to generate sample ions by electron impact, chemical ionization, or charge exchange.

5.2.3.2 *mass analyzer* — a device that utilizes electric and/or magnetic fields to separate charged particles or ions according to their mass-to-charge (m/e) ratios. Examples of mass analyzers include quadrupole, magnetic and/or electric sector, time of flight, and ion traps.

5.2.3.3 *electron multiplier* — a device that detects and amplifies electro-magnetic phenomena such as positive/negative ions.

5.2.4 *back pressure regulator* — a self-contained device, consisting of a mechanical or electrical sensor and control device, commonly used in the semiconductor industry to maintain a constant pressure upstream of the regulator.

5.2.5 *breakthrough* — the point in time when an individual impurity level in the purifier effluent exceeds the level specified by the manufacturer. Typically in the range of 1–100 ppb.

5.2.6 *challenge gas* — a gas mixture containing high levels of gas impurities. Typically, a challenge gas has impurities of between 500 ppm to 1% which is used to shorten the test duration; however, challenges in the range of 1–10 ppm for the impurities is more representative.

5.2.7 *gaseous impurities* — gas phase elements and compounds in the gas stream other than the process or base gas.

5.2.8 *impurity analyzer* — an appropriate analyzer to measure the concentration of desired impurities in a gas stream from the ppm to the percent (%) concentration range.

5.2.9 *inert gas* — a gas, which at ambient conditions, does not react chemically with other materials or chemicals.

5.2.10 *limit of detection (LOD)* — lowest concentration that can be detected by an instrument. LOD is typically defined as three times the standard deviation of the mean noise level (see SEMI F6, lower detectable limit of instrument).

5.2.11 *mass flow controller (MFC)* — a self-contained device, consisting of a mass flow transducer, control valve, and control and signal-processing electronics, commonly used in the semiconductor industry to measure and regulate the mass flow of gas (as defined in SEMI E29).

5.2.12 *pure gas* — an inert gas, minimum purity of 99.9995%, and less than 1 ppb of each impurity that is specified to be removed by the DUT.

5.2.13 *purifier* — generally a catalytic (getter, reactive), resinous, or diatomaceous material within a pressure vessel which removes particulate and/or trace gas impurities from a gas stream (as defined in SEMI F22).

5.2.14 *purifier capacity* — the total quantity of each trace gas impurity that may be sorbed by the purifier media. Defined as liters impurity/liter purifier media.

5.2.15 *regeneration* — the process of reactivating the purifier media.

5.2.16 *test duration* — total time required to complete the test procedure.

5.2.17 *test flow rate* — flow rate through DUT (slpm).

5.2.18 *test pressure* — pressure immediately upstream of the DUT.

5.2.19 *test temperature* — operating temperature of DUT.

5.2.20 *ultratrace analytical instrumentation* — instrumentation that has sufficient sensitivity to

measure all impurities of interest at the specified level of the customer, the ppb or sub-ppb level.

5.2.21 *zero gas* — nitrogen, argon, helium or hydrogen with an estimated level an order of magnitude, or more, lower than the lowest calibration point for each impurity of interest (as defined in SEMI F33).

6 Summary of Method

6.1 This method will allow a user to quantify the impurity removal capacity of a given inert gas purifier for that impurity.

7 Safety Precautions

7.1 This test method may involve hazardous materials, operations, and equipment. The test method does not purport to address the safety considerations associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicability of regulatory limitations before using this method.

7.2 Exhaust from the DUT should be properly vented.

7.3 Only the appropriate gas should be used for purifier testing. Use of the inappropriate gas may cause exothermic reactions and possible explosions.

7.4 Electric discharges or mechanical friction might trigger combustion within a getter. Avoid situations where there is an accumulation of electrostatic charge.

7.5 Purifiers are generally designed for use with impurity levels less than 1% and should not be used to purify air or other inappropriate gases. Contact the manufacturer if there is any question as to the suitability for a particular gas.

7.6 Care should be taken to minimize the purifier's exposure to room air (even filtered air). Room air may chemically react with some purifiers shortening the purifier lifetime. Follow manufacturer's installation procedures.

8 Test Protocol

8.1 Test Conditions

8.1.1 The test should be conducted following manufacturers recommended handling procedures to activate new media or regenerate existing purifier media.

8.1.2 The test is to be conducted at a room temperature maintained between 18°C (64°F) and 26°C (78°F). Environmental temperature fluctuations within this range are not expected to have any measurable effect on the instrumentation used to detect the level of

impurities. Follow instrument manufacturer's operating procedures.

8.1.3 Testing performed at high impurity challenge may alter test results, and may not be appropriate to do for all types of purifiers. Any significant temperature change during the test could have adverse or false effects on the capacity results.

8.1.4 For a mixture of more than 1 impurity, competing reactions may occur between impurities, which may lead to different results.

8.2 Apparatus

8.2.1 Materials

8.2.1.1 *Test Gas* — a mixture of pure gas and challenge gas. Select the appropriate concentration level. For initial studies, a higher concentration range, e.g., 500 ppm to 1% is suggested for each given impurity. For more representative studies done over longer time periods, 1–10 ppm for each given impurity is suggested.

8.2.1.2 *Pressure Regulators* — all wetted internal surfaces, where appropriate, should be made of electropolished 316L stainless steel with an internal surface finish of 0.18 μm (7 μin) R_a and 0.25 μm (10 μin) $R_{a,\text{max}}$, to control system pressures.

8.2.1.3 *Pressure Gauge* — all wetted internal surfaces, where appropriate, should be made of electropolished 316L stainless steel with an internal surface finish of 0.18 μm (7 μin) R_a and 0.25 μm (10 μin) $R_{a,\text{max}}$, to monitor system pressures.

8.2.1.4 *Standard Test Flows* — use appropriate mass flow devices. One MFC with appropriate range of 0–50 slpm for the pure gas is suggested. Various MFCs with appropriate ranges of 0–25 sccm, 0–100 sccm and 0–1 slpm for the challenge gas is suggested.

8.2.1.5 *Tubing* — made of electropolished 316L stainless steel, with an internal surface finish of 0.18 μm (7 μin) R_a and 0.25 μm (10 μin) $R_{a,\text{max}}$, to transport gas.

8.2.1.6 *Fittings* — the appropriate size face-seal fitting is used.

8.2.1.7 *Gaskets* — use metal gaskets for all connections. New gaskets should be used for each new connection. Use of cleanroom gloves is required when handling gaskets and fittings.

8.2.2 Instrumentation

8.2.2.1 An APIMS or other ultratrace analytical instrumentation is used to determine the level of each gaseous impurity exiting the DUT.

8.2.2.2 An impurity analyzer is used to measure higher concentrations of impurities such as found in the test gas.

8.2.2.3 Electronically controlled mass flow controllers are used to accurately blend the impurity challenge level.

8.2.2.4 Data collection equipment is used to gather output from the ultratrace analytical instrumentation.

8.2.2.5 All instruments used should be calibrated regularly, according to manufacturer's specifications.

8.2.3 *Test Set-up and Schematic*

8.2.3.1 Assemble the test setup according to Figure 1. Do not install the DUT until a purge flow is established through MFC1.

8.2.3.2 For the test set-up, pure gas is blended with challenge gas to create a test gas mixture.

8.2.3.3 The DUT is connected, purged per the manufacturer's recommendation, positioned with the appropriate attitude (if required by the manufacturer), and heated (if required by manufacturer) under pure gas flow.

8.2.3.4 Challenge gas flow is introduced and the impurity analyzer measures the impurity levels. If appropriate, the APIMS or other ultratrace analytical instrumentation may be used to measure the test gas while the test gas bypasses the DUT. See Section 9 on Exposure Precautions.

8.2.3.5 Measure and record the test gas concentration for the desired impurity.

8.2.3.6 Following measurement of the test gas, the bypass should be purged with pure gas to ensure impurity removal from the section of the test set-up that is downstream of the DUT.

8.3 *Test Procedures* — Refer to Figure 1.

8.3.1 Use of the impurity analyzer is recommended to protect the APIMS or ultratrace analytical instrumentation from high impurity concentrations which may harm the instrument. The test may be conducted without the impurity analyzer at the risk of exposing the APIMS or ultratrace analytical instrumentation to high impurity concentrations.

8.3.2 *Analytical Instrumentation Setup*

8.3.2.1 Set up and calibrate the analytical instrumentation (APIMS or ultratrace analytical instrumentation and impurity analyzer) according to manufacturer specifications. This includes establishing the appropriate flow rate to the instrument.

8.3.2.2 Acquire zero data to establish the analytical instrumentation baseline and stability prior to starting the test.

8.3.3 Establish flow of pure gas through the manifold bypass:

8.3.3.1 Start with all valves closed except purge gas to analytical instrumentation (V11 and V13 open).

8.3.3.2 Open V1 and adjust R1 to the suggested operating pressure range of 275–415 kPa (40–60 psig).

8.3.3.3 Open V2, V8, V9 and adjust R3 to provide appropriate backpressure for operation of the APIMS or other ultratrace analytical instrumentation. R3 will vent excess gas providing the volume challenge to the DUT. Set MFC1 to the appropriate flow rate. Readjust R1 and R3 to obtain the proper operating conditions.

8.3.4 Monitor drydown of the manifold bypass:

8.3.4.1 Close V11 and Open V10.

8.3.4.2 Purge the bypass manifold until the impurity level is in the range of the APIMS or ultratrace analytical instrumentation.

8.3.4.3 Close V13 and Open V12. Close V10 and Open V11.

8.3.4.4 Purge the bypass manifold until the moisture impurity level at the APIMS or ultratrace analytical instrumentation is below 1.0 ppb.

8.3.5 Re-isolate the APIMS or ultratrace analytical instrumentation:

8.3.5.1 Close V12 and Open V13. Maintain a constant purge to the analytical instrumentation.

8.3.6 Install the DUT, Purging with Pure Gas:

8.3.6.1 Open V6. Remove the DUT inlet face-seal connection. Quickly install the DUT inlet. Remove the DUT outlet face-seal connection and install the DUT outlet. Open V7.

8.3.6.2 Isolate the manifold bypass, directing all flow through the DUT, close V8 and V9.

8.3.6.3 Adjust R1 and R3 until P1 measures the stated purifier operating pressure.

8.3.6.4 Purge the DUT per the manufacturer's recommendation. If required, heat the DUT per manufacturer's recommendation.

8.3.7 Monitor the impurity level at the outlet of the DUT until stable:

8.3.7.1 Initial impurity monitoring may be done with the impurity analyzer. Close V11, open V9 and V10.

8.3.7.2 When the impurity level is in the range of the APIMS or ultratrace analytical instrumentation, Close V13 and Open V12. Close V9 and V10, Open V11.

8.3.7.3 Monitor impurity level until stable.

8.3.8 Initiate Impurity Challenge:

8.3.8.1 Isolate the DUT and APIMS or ultratrace analytical instrumentation. Close V7 and V12, open V13.

8.3.8.2 Open V3 and adjust R2 to the suggested operating pressure range of 275–415 kPa (40–60 psig). Open V5 and set MFC2 to the desired flow. (See Appendix 1 to determine the desired flow for MFC2).

8.3.8.3 Verify test gas impurity. Open V4, V8, and V10 and then close V5 and V11, supplying test gas to the impurity analyzer. Adjust R2 as necessary to maintain appropriate pressure for supplying MFC2. Monitor test gas impurity until stable and verified to theoretical impurity value (See Appendix 1).

8.3.8.4 Initiate test. Direct input test gas through the DUT to the APIMS or ultratrace impurity analyzer, Open V6 and V7. Open V12 and close V13. Isolate impurity analyzer, Close V8 and V10. Open V11.

8.3.8.5 Zero test gas flow totalizers (Pure Gas MFC1 and challenge gas through MFC2) beginning Purifier Capacity Test.

8.3.9 Determination of purifier capacity:

8.3.9.1 The test will require several weeks or months depending on the purifier's capacity for retention of and the concentration of the impurity.

8.3.9.2 The test may be done on newly activated media or may be done using existing media that has been regenerated. The test may be destructive to the DUT.

8.3.9.3 Constant monitoring of the purifier effluent over the duration of the test is preferred. However, periodic monitoring, e.g. every few hours is sufficient.

8.3.9.4 The test gas can be periodically monitored at the impurity analyzer by closing V11 and opening V8 and V10. If significant, ensure that flow volume to impurity analyzer during this time is subtracted from total volume input through DUT. In addition, instrument calibration should be monitored as recommended by the manufacturer.

8.3.9.5 Monitor trends in outlet purity until the breakthrough point for the impurity is detected. Time, liters of gas, and outlet purity should be recorded.

8.4 Repeat test for each new impurity to be analyzed using a new or regenerated DUT.

9 Exposure Precautions

9.1 The APIMS or ultratrace analytical instrument should not be exposed to high levels of impurities. After installation of the purifier, it should be purged well, per the manufacturer's recommendation. A typical recommendation might be to purge a minimum of 150 bed volumes before directing the flow to the APIMS.

10 Calculation of Purifier Capacity

10.1 Use the following formula to calculate Purifier Capacity:

10.2 $(\text{Test Duration (min)}) \times (\text{Test Flow Rate slpm}) = \text{Total Liters Test Gas (TLTG)}$

10.3 $\text{Impurity(liters)} = (\text{TLTG}) \times \text{ppm} \times 10^{-6}$

10.4 $\text{Purifier Capacity} = (\text{Total Impurity (liters)})/(\text{Purifier Volume(liters)})$

10.5 Purifier Capacity is reported as total liters of each given impurity per liter purifier, and is a dimensionless number.

11 Reporting Results

11.1 The following test conditions should be reported:

11.1.1 Date and time of test,

11.1.2 Operator,

11.1.3 Pure gas flow rate (slpm),

11.1.4 Challenge gas flow rate (sccm),

11.1.5 Test pressure kPa (psig or psia),

11.1.6 DUT operating temperature (° C),

11.1.7 Purifier manufacturer, model, and serial number, and volume,

11.1.8 Ultratrace analytical instrumentation used,

11.1.9 Test gas impurities and levels, and

11.1.10 Calibration certificates for the mass flow devices, pressure gauges and ultratrace analytical instrumentation.

12 Related Documents

SEMI F30 — Start-up and Verification of Purifier Performance Testing for Trace Gas Impurities and Particles at an Installation Site

SEMI F43 — Test Method for Determination of Particle Contribution by Point-Of-Use Purifiers

NOTE 3: Unless otherwise indicated, all documents cited shall be the latest published versions.

APPENDIX 1

MFC SIZING

NOTE: The material in this appendix is an official part of SEMI F67 and was approved by full letter ballot procedures on August 27, 2001.

A1-1 To determine purifier efficiency, the concentration of impurity entering the purifier and the quantity of impurity downstream of the purifier must be determined.

A1-2 Determine the manufacturer's recommended flow rate for the purifier. As an example, assume a flow rate of 5 slpm.

A1-3 Determine the flow rate of challenge gas required. As an example, 5 sccm of a 1% challenge gas would be blended into 4,995 sccm of pure gas to generate a 10 ppm test gas.

$$\text{TestGas} = \frac{10,000 \text{ PPM} \times 5 \text{ sccm}}{5 \text{ sccm} + 4,995 \text{ sccm}} = 10 \text{ PPM}$$

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SEMI F68-1101

TEST METHOD FOR DETERMINING PURIFIER EFFICIENCY

This test method 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 27, 2001. Initially available at www.semi.org September 2001; to be published November 2001.

1 Purpose

1.1 The purpose of this document is to define a test method to quantify the efficiency of a purifier for removal of an active gaseous impurity from a matrix gas.

2 Scope

2.1 To determine the efficiency of a gas purifier to remove a given impurity species. Efficiency tests are performed by adding ppm levels of gaseous impurities to a pure matrix gas and monitoring the effluent of the test purifier for active impurity species. Tests are done at supplier recommended flow rate, operating temperature and pressure.

2.2 To establish a method of determining instantaneous purifier efficiency.

2.3 The test method applies to point of use (POU) and large scale purifiers.

2.4 This method is for UHP efficient removal of low level contaminants.

2.5 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 and safety health practices and determine the applicability of regulatory limitations prior to use.

3 Limitations

3.1 The inherent limitation to this method is the limit of detection (LOD) of the analytical instrument employed by the user.

3.2 This test method can only be used to compare purifier efficiency results if the user application for flow rate, pressure, and temperature are the same as the test conditions. Different user and/or different operating conditions may result in different purifier performance results.

3.3 In testing mixtures of impurities, some impurities may influence the efficiency results of other impurities. Discussion with the manufacturer is highly recommended prior to testing.

3.4 The test method does not apply to particulates.

3.5 This test method will provide efficiency information only for impurities that are used in the challenge gas.

4 Referenced Standards

4.1 SEMI Standards

SEMI E29 — Standard Terminology for the Calibration of Mass Flow Controllers and Mass Flow Meters

SEMI F6 — Guide for Secondary Containment of Hazardous Gas Piping Systems

SEMI F22 — Guide for Gas Distribution Systems

SEMI F33 — Method for Calibration of Atmospheric Pressure Ionization Mass Spectrometer (APIMS)

4.2 ANSI Standards¹

ANSI B46.1 — Surface Texture (Surface Roughness, Waviness, and Lay)

NOTE 1: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 Abbreviations and Acronyms

5.1.1 APIMS — atmospheric pressure ionization mass spectrometer

5.1.2 °C — degrees Celsius

5.1.3 DUT — device under test

5.1.4 °F — degrees Fahrenheit

5.1.5 in — inch

5.1.6 kPa — kiloPascal

5.1.7 LOD — limit of detection

5.1.8 m — meter

5.1.9 MFC — mass flow controller

5.1.10 NMHC — non methane hydrocarbons

5.1.11 POU — point of use

5.1.12 ppb — parts per billion, volume basis

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

5.1.13 *ppm* — parts per million, volume basis

5.1.14 *psi* — pounds per square inch

5.1.15 *psia* — pounds per square inch absolute

5.1.16 *psig* — pounds per square inch gauge

5.1.17 R_a — surface roughness average (as defined in ANSI B46.1)

5.1.18 $R_{a,max}$ — surface roughness maximum (as defined in ANSI B46.1)

5.1.19 *s* — second

5.1.20 *sccm* — standard cubic centimeters per minute

5.1.21 *slpm* — standard liters per minute

5.1.22 *UHP* — ultra high purity

5.2 Definitions

5.2.1 *activation* — the process of initially preparing the purifier media to be chemically reactive with gas impurities.

5.2.2 *activation temperature* — temperature at which DUT was initially prepared.

5.2.3 *atmospheric pressure ionization mass spectrometer (APIMS)* — an instrument consisting of an atmospheric pressure ion source where gas phase impurities are ionized via charge exchange reactions with the bulk gas. These ions are directed into a vacuum chamber where they are then separated by a mass analyzer and detected by an electron multiplier.

5.2.3.1 *ion source* — the section of a mass spectrometer used to generate sample ions by electron impact, chemical ionization, or charge exchange.

5.2.3.2 *mass analyzer* — a device that utilizes electric and/or magnetic fields to separate charged particles or ions according to their mass-to-charge (m/e) ratios. Examples of mass analyzers include quadrupole, magnetic and/or electric sector, time of flight, and ion traps.

5.2.3.3 *electron multiplier* — a device that detects and amplifies electro-magnetic phenomena such as positive/negative ions.

5.2.4 *back pressure regulator* — a self-contained device, consisting of a mechanical or electrical sensor and control device, commonly used in the semiconductor industry to maintain a constant pressure upstream of the regulator.

5.2.5 *breakthrough* — the point in time when an individual impurity level in the purifier effluent exceeds the level specified by the manufacturer. Typically in the range of 1–100 ppb.

5.2.6 *challenge gas* — a gas mixture containing high levels of gas impurities. Typically, a challenge gas has impurities of between 500 ppm to 1% which is used to shorten the test duration; however, challenges in the range of 1–10 ppm for the impurities is more representative.

5.2.7 *efficiency* — a measure of the ability of a purifier to remove active impurities from a matrix gas stream. It is calculated as the ratio of the difference between the inlet concentration and the concentration of impurity leaving the purifier to the concentration of impurity entering the purifier.

5.2.8 *gaseous impurities* — gas phase elements and compounds in the gas stream other than the process or base gas.

5.2.9 *impurity analyzer* — an appropriate analyzer to measure the concentration of desired impurities in a gas stream from the ppm to the percent (%) concentration range.

5.2.10 *inert gas* — a gas, which at ambient conditions, does not react chemically with other materials or chemicals.

5.2.11 *limit of detection (LOD)* — lowest concentration that can be detected by an instrument. LOD is typically defined as three times the standard deviation of the mean noise level (see SEMI F6, lower detectable limit of instrument).

5.2.12 *mass flow controller (MFC)* — a self-contained device, consisting of a mass flow transducer, control valve, and control and signal-processing electronics, commonly used in the semiconductor industry to measure and regulate the mass flow of gas (as defined in SEMI E29).

5.2.13 *pure gas* — an inert gas, minimum purity of 99.9995%, and less than 1 ppb of each impurity that is specified to be removed by the DUT.

5.2.14 *purifier* — generally a catalytic (getter, reactive), resinous, or diatomaceous material within a pressure vessel which removes particulate and/or trace gas impurities from a gas stream (as defined in SEMI F22).

5.2.15 *purifier capacity* — the total quantity of each trace gas impurity that may be sorbed by the purifier media. Defined as liters impurity/liter purifier media.

5.2.16 *regeneration* — the process of reactivating the purifier media.

5.2.17 *test duration* — total time required to complete the test procedure.

5.2.18 *test flow rate* — flow rate through DUT (slpm).

5.2.19 *test pressure* — pressure immediately upstream of the DUT.

5.2.20 *test temperature* — operating temperature of DUT.

5.2.21 *ultratrace analytical instrumentation* — instrumentation that has sufficient sensitivity to measure all impurities of interest at the specified level of the customer, the ppb or sub-ppb level.

5.2.22 *zero gas* — nitrogen, argon, helium or hydrogen with an estimated level an order of magnitude, or more, lower than the lowest calibration point for each impurity of interest (as defined in SEMI F33).

6 Summary of Method

6.1 This method will allow a user to quantify the impurity efficiency of a point-of-use (POU) or large scale purifier.

7 Safety Precautions

7.1 This test method may involve hazardous materials, operations, and equipment. The test method does not purport to address the safety considerations associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and determine the applicability of regulatory limitations before using this method.

7.2 Exhaust from the DUT should be properly vented.

7.3 Only the appropriate gas should be used for purifier testing. Use of inappropriate gases may cause exothermic reactions and possible explosions.

7.4 Electric discharges or mechanical friction might trigger combustion within a getter. Avoid situations where there is an accumulation of electrostatic charge.

7.5 Purifiers are generally designed for use with impurity levels less than 1% and should not be used to purify air or other inappropriate gases. Contact the manufacturer if there is any question as to the suitability for a particular gas.

7.6 Care should be taken to minimize the purifier's exposure to room air (even filtered air). Room air may chemically react with some purifiers shortening the purifier lifetime. Follow manufacturer's installation procedures.

8 Test Protocol

8.1 Test Conditions

8.1.1 The test should be conducted following manufacturer's recommended handling procedures to activate new media or regenerate existing purifier media.

8.1.2 The test is to be conducted at a room temperature maintained between 18°C (64°F) and 26°C (78°F). Environmental temperature fluctuations within this range are not expected to have any measurable effect on the instrumentation used to detect the level of impurities. Follow instrument manufacturer's operating procedures.

8.2 Apparatus

8.2.1 Materials

8.2.1.1 *Test Gas* — a mixture of pure gas and challenge gas. The mixture should contain gaseous impurities of between 1 ppm and 10 ppm.

8.2.1.2 *Pressure Regulators* — all wetted internal surfaces, where appropriate, should be made of electropolished 316L stainless steel with an internal surface finish of 0.18 μm (7 μin) R_a and 0.25 μm (10 μin) $R_{a,\text{max}}$, to control system pressures.

8.2.1.3 *Pressure Gauge* — all wetted internal surfaces, where appropriate, should be made of electropolished 316L stainless steel with an internal surface finish of 0.18 μm (7 μin) R_a and 0.25 μm (10 μin) $R_{a,\text{max}}$, to monitor system pressures.

8.2.1.4 *Standard Test Flows* — use appropriate mass flow devices. One MFC with appropriate range of 0–50 slpm for the pure gas is suggested. Various MFCs with appropriate ranges of 0–25 sccm, 0–100 sccm and 0–1 slpm for the challenge gas are suggested.

8.2.1.5 *Tubing* — made of electropolished 316L stainless steel, with an internal surface finish of 0.18 μm (7 μin) R_a and 0.25 μm (10 μin) $R_{a,\text{max}}$, to transport gas.

8.2.1.6 *Fittings* — the appropriate size face-seal fitting is used.

8.2.1.7 *Gaskets* — use metal gaskets for all connections. New gaskets should be used for each new connection. Use of cleanroom gloves is required when handling gaskets and fittings.

8.2.2 Instrumentation

8.2.2.1 An APIMS or other ultratrace analytical instrumentation is used to determine the level of each gaseous impurity exiting the DUT.

8.2.2.2 An impurity analyzer is used to measure higher concentrations of impurities such as found in the test gas.

8.2.2.3 Electronically controlled mass flow controllers are used to accurately blend the impurity challenge level.

8.2.2.4 Data collection equipment is used to gather output from the ultratrace analytical instrumentation.

8.2.2.5 All instruments used should be calibrated regularly, according to manufacturer specifications.

8.2.3 *Test Setup and Schematic*

8.2.3.1 Assemble the test setup according to Figure 1. For a large scale system which may include components such as flow meters, pressure regulation and indication, and bypass loops around the purifying media(s), the test setup may be modified accordingly in order to use these built in attributes while adhering to the procedural steps. Do not install the DUT until a purge flow is established through MFC1.

8.2.3.2 Pure gas is blended with challenge gas to create a test gas mixture containing approximately 1–10 ppm of gaseous impurities.

8.2.3.3 The DUT is connected, purged per the manufacturer's recommendation, positioned with the appropriate attitude (if required by the manufacturer), and heated (if required by manufacturer) under pure gas flow.

8.2.3.4 Challenge gas flow is introduced and the impurity analyzer measures the impurity levels. If appropriate, the APIMS or other ultratrace analytical instrumentation may be used to measure the test gas while the test gas bypasses the DUT. See Section 9 on Exposure Precautions.

8.2.3.5 Measure and record the test gas concentration for the desired impurity.

8.2.3.6 Instantaneous impurity efficiencies may be calculated at any point by knowing the level of each impurity entering and exiting the purifier.

8.3 *Test Procedures* — Refer to Figure 1.

8.3.1 Use of the impurity analyzer is recommended to protect the APIMS or other ultratrace analytical instrumentation from impurity spikes which may harm the instrument. The test may be conducted without the impurity analyzer at the risk of such spikes.

8.3.2 *Analytical Instrumentation Setup*

8.3.2.1 Set up and calibrate the analytical instrumentation (APIMS or ultratrace analytical instrumentation and impurity analyzer) according to manufacturer specifications. This includes but is not limited to establishing the appropriate flow rates to the instruments.

8.3.2.2 Acquire zero data to establish the instrumentation baseline and stability prior to starting the test.

8.3.3 Establish flow of pure gas through the manifold bypass:

8.3.3.1 Start with all valves closed except purge gas to analytical instrumentation (V11 and V13 open).

8.3.3.2 Open V1 and adjust R1 to the suggested operating pressure range of 275–415 kPa (40–60 psig).

8.3.3.3 Open V2, V8, V9 and adjust R3 to provide appropriate backpressure for operation of the APIMS or other ultratrace analytical instrumentation. R3 will vent excess gas providing the volume challenge to the DUT. Set MFC1 to the appropriate flow rate.

8.3.4 Monitor drydown of the manifold bypass:

8.3.4.1 Close V11 and Open V10.

8.3.4.2 Purge the bypass manifold until the impurity level is in the range of the APIMS or ultratrace analytical instrumentation.

8.3.4.3 Close V13 and Open V12.

8.3.4.4 Purge the bypass manifold until the moisture impurity level at the APIMS or ultratrace analytical instrumentation is below 1.0 ppb.

8.3.5 Re-isolate the APIMS or ultratrace analytical instrumentation:

8.3.5.1 Close V12 and Open V13. Close V10 and open V11. Maintain a constant purge to the analytical instrumentation.

8.3.6 Install the DUT, purging with Pure Gas (may not be necessary, as installed in the test set-up for large scale systems):

8.3.6.1 Open V6. Remove the DUT (purifier) inlet face-seal connection. Quickly install the DUT inlet. Remove the DUT outlet face-seal connection and install the DUT outlet. Open V7.

8.3.6.2 Isolate the manifold bypass, directing all flow through the DUT, close V8 and V9.

8.3.6.3 Adjust R1 and R3 until P1 measures the stated purifier operating pressure.

8.3.6.4 Purge the DUT (purifier) per the manufacturer's recommendation. If required, heat the DUT (purifier) per manufacturer's recommendation.

8.3.7 Monitor the impurity level at the outlet of the DUT until stable:

8.3.7.1 Initial impurity monitoring may be done with the impurity analyzer. Close V12, Open V9 and V10. Close V11.

8.3.7.2 When the impurity level is in the range of the APIMS or ultratrace analytical instrumentation, Close V13 and Open V12. Close V9 and V10, Open V11.

8.3.7.3 Monitor impurity level until stable.

8.3.8 Initiate Impurity Challenge:

8.3.8.1 Isolate the DUT and APIMS or ultratrace analytical instrumentation. Close V7 and V12, open V13.

8.3.8.2 Open V3 and adjust R2 to the suggested operating pressure range of 275–415 kPa (40–60 psig). Open V5 and set MFC2 to the desired flow. (See Appendix 1 to determine the desired flow for MFC2).

8.3.8.3 Verify test gas impurity. Close V5 and V11, Open V4, V8, and V10 supplying test gas to the impurity analyzer. Monitor test gas impurity until stable and verified to theoretical impurity value (See Appendix 1).

8.3.8.4 Initiate test. Isolate impurity analyzer, Close V8 and V10, Open V11. Input test gas through the DUT to the APIMS or ultratrace impurity analyzer, Open V6 and V7. Close V13 and Open V12.

8.3.8.5 Monitor and record the outlet impurity level until stable.

8.3.8.6 If necessary, repeat the test for a different challenge gas mixture.

8.3.9 Determination of instantaneous purifier efficiency:

8.3.9.1 The test may be done on newly activated media or may be done using existing media that has been regenerated. The test may be destructive to the DUT (purifier).

8.3.9.2 Repeat the test as necessary to provide a statistically valid number of test results. A discussion of what is statistically valid is beyond the scope of this document.

9 Exposure Precautions

9.1 The APIMS or ultratrace analytical instrument should not be exposed to high levels of impurities. After installation of the purifier, it should be purged well, per the manufacturer's recommendation. A typical recommendation might be to purge a minimum of 150 bed volumes before directing the flow to the APIMS.

9.2 Alternative instrumentation should be used to measure the challenge gas when the concentration of

the challenge gas is greater than the normal calibration range of the APIMS or other ultratrace analytical instrumentation.

10 Calculation of Instantaneous Purifier Efficiency

10.1.1.1 Use the following formula to calculate the Instantaneous Purifier Efficiency:

$$\%eff = \left(1 - \frac{\text{outlet impurity}}{\text{inlet impurity}}\right) \times 100$$

Example: Let impurity outlet = 10 ppb and impurity inlet = 10 ppm,

$$\%eff = \left(1 - \frac{10}{10,000}\right) \times 100 = 99.9$$

11 Reporting Results

11.1 The following test conditions should be reported:

11.1.1 Date and time of test,

11.1.2 Operator,

11.1.3 Pure gas flow rate (slpm),

11.1.4 Challenge gas flow rate (sccm),

11.1.5 Test pressure kPa (psig or psia),

11.1.6 DUT operating temperature (° C),

11.1.7 Purifier manufacturer, model, and serial number, and volume,

11.1.8 Ultratrace analytical instrumentation used,

11.1.9 Test gas impurities and levels (Table 1), and

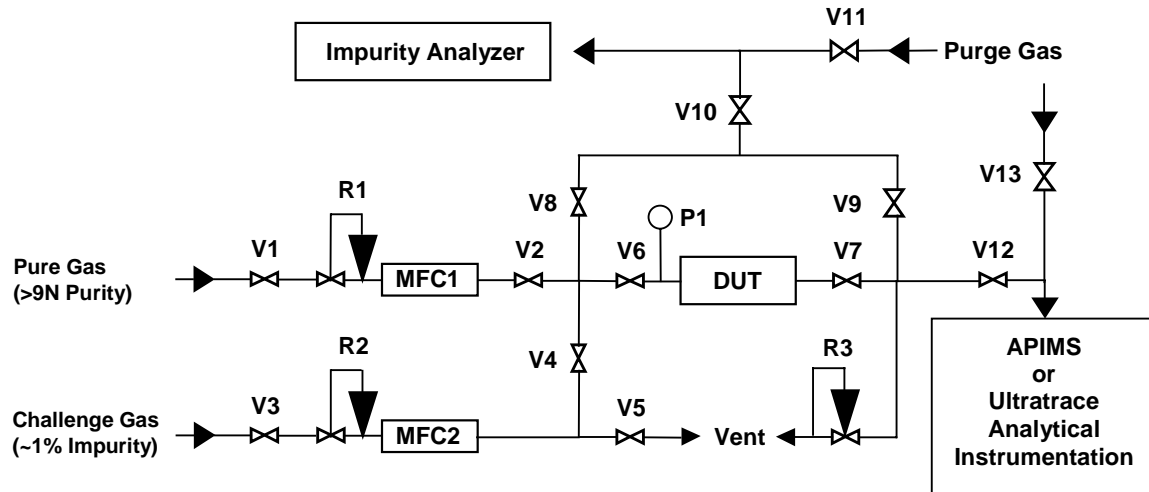
11.1.10 Calibration certificates for the mass flow devices, pressure gauges and ultratrace analytical instrumentation.

12 Related Documents

SEMI F30 — Start-up and Verification of Purifier Performance Testing for Trace Gas Impurities and Particles at an Installation Site.

SEMI F43 — Test Method for Determination of Particle Contribution by Point-Of-Use Purifiers.

NOTE 2: Unless otherwise indicated, all documents cited shall be the latest published versions.



Legend

DUT = Device Under Test	V6 = DUT Inlet Isolation Valve
P1 = Test Pressure	V7 = DUT Outlet Isolation Valve
R1 = Pure Gas 0–100 psig Regulator	V8 = DUT Bypass Inlet Isolation Valve
R2 = Challenge Gas 0–100 psig Regulator	V9 = DUT Bypass Outlet Isolation Valve
R3 = Vent Gas 0–100 psig Back Pressure Regulator	V10 = Impurity Analyzer Sample Isolation Valve
V1 = Pure Gas Source Isolation Valve	V11 = Impurity Analyzer Purge Gas Isolation Valve
V2 = Pure Gas System Isolation Valve	V12 = APIMS/UAI Sample Isolation Valve
V3 = Challenge Gas Source Isolation Valve	V13 = APIMS/UAI Purge Gas Isolation Valve
V4 = Challenge Gas System Isolation Valve	MFC1 = Pure Gas Mass Flow Controller
V5 = Challenge Gas Vent Isolation Valve	MFC2 = Challenge Gas Mass Flow Controller

Figure 1
Suggested Point of Use Purifier Efficiency Test Setup

Table 1 Efficiency Of DUT Parameters

EFFICIENCY OF DUT						
Impurity	Pure Gas Flow (slpm)	Challenge Flow (sccm)	Inlet Conc (ppm)	Outlet Conc (ppb)	Zero Gas (ppb)	Analyzer
CH ₄						
H ₂						
O ₂						
N ₂						
CO ₂						
NMHC						

APPENDIX 1

MFC SIZING

NOTE: The material in this appendix is an official part of SEMI F68 and was approved by full letter ballot procedures on August 27, 2001.

A1-1 To determine purifier efficiency, the concentration of impurity entering the purifier and the quantity of impurity downstream of the purifier must be determined.

A1-2 Determine the manufacturer's recommended flow rate for the purifier. As an example, assume a flow rate of 5 slpm.

A1-3 Determine the flow rate challenge gas required. As an example, 5 sccm of a 1% challenge gas would be blended into 4,995 sccm of pure gas to create at 10 ppm test gas.

$$\text{Test Gas} = \frac{10,000 \text{ PPM} \times 5 \text{ sccm}}{5 \text{ sccm} + 4,995 \text{ sccm}} = 10 \text{ PPM}$$

As a large scale system, with a flow of 1,000 slpm, a flow of 1 slpm of challenge gas (1% impurity) is required to create a 10 ppm challenge.

$$\text{Test Gas} = \frac{10,000 \text{ PPM} \times 1 \text{ slpm}}{1 \text{ slpm} + 999 \text{ slpm}} = 10 \text{ PPM}$$

NOTICE: SEMI makes no warranties or representations as to the suitability of the standard 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 F69-0302

TEST METHODS FOR TRANSPORT AND SHOCK TESTING OF GAS DELIVERY SYSTEMS

This test method 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 November 27, 2001. Initially available at www.semi.org December 2001; to be published March 2002.

1 Purpose

1.1 This document provides test methods for qualifying the mechanical integrity of gas delivery systems through vibration and shock testing.

2 Scope

2.1 The test methods recommended herein provide for vibration (transport simulation) and shock testing of gas delivery systems for semiconductor processing.

2.2 The test methods recommended herein apply to gas delivery systems not crated or packaged for shipment. Specifically, the test methods are to be applied to the assembled and interconnected gas delivery components and their associated mounting panel (back plane), with or without a sheet metal enclosure.

2.3 For the purpose of this guideline, transportation vibration, and its simulation, are expectedly more severe than in-use vibrational levels. Thus the transportation simulation test is considered acceptable to assess mechanical integrity adequate for both shipment and life-cycle vibrational stress of the gas delivery systems.

2.4 The intent of the shock test is to provide further assessment of equipment malfunction that may result from shocks experienced during unpacking, installation, or use in the field.

2.5 Successful completion of the tests recommended herein is a recommended metric of mechanical integrity for gas delivery systems architecture, design, and assembly techniques.

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

3 Limitations

3.1 This document, as a guide, does not provide detailed information sufficient for conducting the tests. It is the responsibility of the user and testing entity to procure a copy of the referenced test procedures from the issuing organization(s).

3.2 The test methods recommended herein are intended to evaluate gas delivery systems architectures, design principles, and assembly methodologies, not individual production gas delivery systems.

3.3 The functional components of gas delivery systems, for example mass flow controllers and pressure transducers, may be adversely affected by the forces seen during these tests. Such components may require recalibration after testing.

4 Referenced Standards

NOTE 1: As listed or revised, all documents cited shall be the latest publications of adopted standards.

4.1 SEMI Standards

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

4.2 Military Standards¹

MIL-STD-810 — Environmental Engineering Considerations and Laboratory Tests

5 Terminology

5.1 *bag leak test* — A helium leak testing procedure in which the system undergoing leak test is placed in a helium-filled plastic bag while connected to a functional helium leak detector.

5.2 *g* — A unit of force equal to that exerted by gravity upon a mass in equilibrium on the earth's surface. Expressed in Newtons (kg-m/sec.²).

5.3 *leak tight* — Having a helium leak rate no greater than that specified by the customer or end-user.

6 Vibration Testing

NOTE 2: Vibration testing should be performed in its entirety before continuing to the shock testing.

6.1 In general, conduct vibrational testing according to MIL-STD-810, Part Two, Laboratory Test Method 514.5, Procedure 1, Category 4, for 3 hours in each axis (a total of 9 hours). This will subject the gas delivery

¹ DODSSP, Building 4 / Section D, 700 Robbins Avenue, Philadelphia, PA 19111-5094

systems to the equivalent physical stress of a 4828 kilometer (3000 mile) motor freight shipment over improved or paved highways. Because vibration tables are commonly single-axis devices, the gas delivery systems will be subjected to single-axis random vibrational frequencies in the range of 5—500 Hz, sequentially, in each of its three axes.

6.2 Prior to initiating the test, examine the gas delivery systems for physical defects and document the results.

6.3 Prior to initiating the test, thoroughly prepare the gas delivery systems for testing. This includes, but is not limited to, insuring all fasteners and sealing mechanisms are tightened to manufacturers' or design specifications.

6.4 The MIL-STD-810 transportation test recommended herein provides for different acceleration levels for each of the 3 axes of the device under test. Because the majority of gas delivery systems are shipped in a vertical orientation, this convention is maintained for the purpose of the transportation simulation tests. Thus the vertical orientation would have the gas delivery system mounted vertically with respect to the vibration table top (see Figure 2), the longitudinal orientation would have the gas delivery system mounted flat to the vibration table top (see Figure 1), and the transverse orientation would have the gas delivery system mounted on its side, against the vibration table top (see Figure 3). Note that the terms "vertical," "longitudinal," and "transverse," are contextual to motor freight transport, and receive different acceleration levels, within MIL-STD-810. A simplified and conservative approach to applying MIL-STD-810 involves applying the highest acceleration level, associated with the vertical axis, to all three orientations as illustrated in Figures 1–3.

6.5 Mount the gas delivery system, in any of its untested axes, securely to the vibration table. See Figures 1–3.

6.6 Prior to initiating the test at the test site, make certain that the gas delivery system is leak tight using inboard test procedures per SEMI F1. A bag leak test method is recommended in conjunction with this test.

6.7 Initiate and run for 3 hours the vibration test corresponding to the orientation of the gas delivery system.

6.8 At the conclusion of the test, examine the gas panel for physical defects and document the results

6.9 At the conclusion of the test, leak test the gas delivery system using inboard leak procedures per SEMI F1. A bag leak test method is recommended in conjunction with this test. Alternatively, by agreement with the customer, all leak testing may be withheld until completion of all shock and vibration testing. This presumes specific failure mode information is not required.

6.10 Repeat Sections 6.5 through 6.9 until all 3 axes are tested.

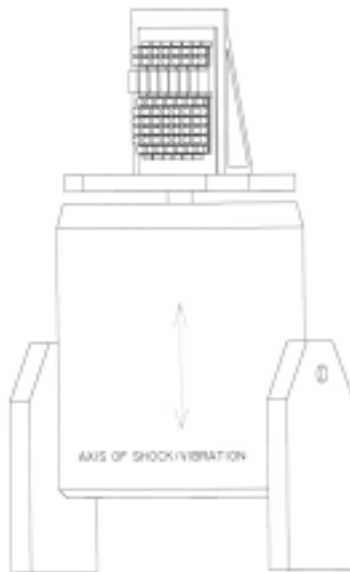
6.11 The post-test report should include, at minimum, the following:

- (1) Pre-test examination results.
- (2) Pre-test leak rates.
- (3) Summary and chronology of test events, test interruptions, and test failures.
- (4) All vibration measurement data.
- (5) Post-test examination results.
- (6) Post-test leak rates.



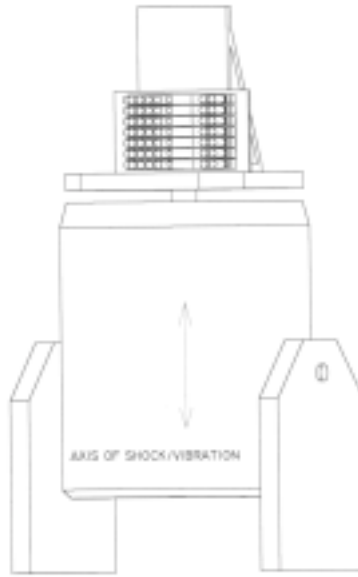
NOTE 1: Plane of gas sticks is parallel to shock/vibe table and transverse to test input.

Figure 1
Horizontal Orientation



NOTE 1: Plane and axes of gas sticks are normal to shock/vibe table and parallel to test input.

Figure 2
Vertical Orientation



NOTE 1: Plane of gas sticks is normal to shock/vibe table and axes of sticks are transverse to test input.

Figure 3
Lateral Orientation

7 Shock Testing

7.1 In general, conduct shock testing according to MIL-STD-810, Part Two, Laboratory Test Method 516.5, Procedure 1 (Functional Shock). This test will subject the gas delivery system to a peak acceleration of 40 g's employing a terminal peak sawtooth shock pulse.

7.2 The test should be conducted in each of the gas delivery system's three axes as illustrated in Figures 1–3.

7.3 Prior to initiating the test, examine the gas delivery system for physical defects and document the results.

7.4 Prior to initiating the test, thoroughly prepare the gas delivery system for testing. This includes, but is not limited to, insuring all fasteners and sealing mechanisms are tightened to manufacturers' or design specifications.

7.5 Prior to initiating the test, make certain that the gas delivery system is leak tight using inboard test procedures per SEMI F1. A bag leak test method is recommended in conjunction with this test.

7.6 Secure the gas delivery system under test to the shock table in one of its three orientations: horizontal, vertical, or lateral. The gas delivery system should be fastened directly to the table with no cushion or other

intermediary between the two. A rigid mounting is desirable, taking caution not to introduce stress to the gas delivery system.

7.7 Conduct the shock test.

7.8 At the conclusion of the test, examine the gas panel for physical defects and document the results.

7.9 If, as a result of the test, an apparent physical failure occurred that would likely prevent the safe operation of the gas panel and/or would likely compromise the leak tightness of the gas panel, the physical failure should be corrected, the gas panel retested for leak tightness, and the test repeated.

7.10 At the conclusion of the test, leak test the gas delivery system using inboard leak procedures per SEMI F1. A bag leak test method is recommended in conjunction with this test. Alternatively, by agreement with the customer, all leak testing may be withheld until completion of all shock and vibration testing. This presumes specific failure mode information is not required.

7.11 Repeat 7.6 through 7.10 for a second orientation.

7.12 Repeat 7.6 through 7.10 for the third and final orientation.

7.13 The post-test report should include, at minimum, the following:

- 1) Pre-test examination results.
- 2) Pre-test leak rate.
- 3) Summary and chronology of test events, test interruptions, and test failures.
- 4) All shock measurement data, including that of any accelerometers mounted to the gas delivery system.
- 5) Post-test examination results for all three axes.
- 6) Post-test leak rate for all three shock tests.

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.

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SEMI F70-0302

TEST METHOD FOR DETERMINATION OF PARTICLE CONTRIBUTION OF GAS DELIVERY SYSTEM

This test method was technically approved by the Global Facilities Committee and is the direct responsibility of the Japanese Facilities Committee. Current edition approved by the Japanese Regional Standards Committee on January 11, 2002. Initially available at www.semi.org January 2002; to be published March 2002.

1 Purpose

1.1 The purpose of this document is to provide a standardized methodology and procedure for measuring the particle contribution performance of a gas delivery system in terms of number of particles added to gas flowing through the system. This standardized procedure is intended to be used commonly by the component suppliers, gas suppliers, equipment suppliers, and users.

2 Scope

2.1 This test method applies to all types of surface mount and conventional gas delivery systems used in semiconductor manufacturing facilities and comparable research and development areas.

2.2 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 limitations prior to use.

3 Limitations

3.1 All components must meet quality requirements as established and controlled by manufacturers prior to testing (e.g., dimensional, functional, etc.).

3.2 Care should be exercised in handling of components to maintain manufacturer's specifications.

4 Referenced Standards

NOTE 1: As listed or revised, all documents cited shall be the latest publications of adopted standards.

4.1 ISO Standards¹

ISO 14644-1 — Cleanrooms and Associated Controlled Environments – Part 1: Classification of air cleanliness

4.2 JIS Standards²

JIS B 9921 — Light Scattering Automatic Particle Counter

5 Terminology

5.1 Abbreviations and Acronyms

5.1.1 *CNC* — condensation nucleus counter

5.1.2 *LPC* — laser particle counter

5.1.3 *MFC* — mass flow controller

5.1.4 *MFM* — mass flow meter

5.1.5 *slm* — standard liters per minute, the gas volumetric flow rate measured in liters per minute at 0°C and 1 atm.

5.2 Definitions

5.2.1 *background counts* — particle counts contributed by the test apparatus (including false counts) with the spool piece in the place of the test object as function of particle size.

5.2.2 *counting efficiency* — the ratio of the particle concentration calculated from the particle counts to the actual particle concentration in the sampled gas for particles equal to or larger than a given particle size.

5.2.3 *design flow rate* — flow rate normally applied to the gas delivery system.

5.2.4 *false counts* — particle counts contributed by electrical noise or by other events and not particles in the sampled gas.

5.2.5 *gas delivery system* — a system installed in semiconductor manufacturing equipment to supply process and carrier gases to reactors, which typically consists of tubing, fittings, valves, filters, mass flow controllers and regulators; can be surface mount or conventional system.

5.2.6 *minimum counting particle diameter* — a predefined minimum diameter of particles to be counted in this test method.

¹ ISO Central Secretariat, 1, rue de Varembe, Case postale 56, CH-1211 Genève 20, Switzerland, website: www.iso.ch

² Japanese Standards Association, 1-24, Akasaka, 4-Chome, Minato-ku, Tokyo 107 Japan

5.2.7 *particle counts* — a counted value from a laser particle counter (LPC) or condensation nucleus counter (CNC) shown for particles or larger than or equal to the minimum counting particle diameter.

5.2.8 *sample flow rate* — the volumetric flow rate through the particle counter.

5.2.9 *spool piece* — a null component consisting of a straight piece of electropolished tubing or like object and appropriate fittings used in place of the test system to establish the background.

5.2.10 *test flow rate* — the volumetric flow rate through the test system

6 Required Apparatus

6.1 *Filter* — Filters with the following performance specified by the manufacturer.

<i>Characteristic</i>	<i>Performance</i>	<i>Condition</i>
<i>Filtration Efficiency</i>	9 log	particle size: 0.02 μ m
<i>Flow Rate</i>	300 slm	inlet pressure: 0.34 MPa

6.2 *Tubing Material* — 1/4 inch or 3/8 inch SUS 316L electropolished tubing or better.

6.3 *Particle Counter* — The particle counter may be either LPC or CNC of which the minimum counting particle diameter is 0.1 μ m or smaller, and has the following performance as specified by JIS B 9921.

6.3.1 *Counting Efficiency*

<i>Diameter of Polystyrene Latex Standard Particle</i>	<i>Counting Efficiency</i>
<i>Minimum countable particle diameter of the counter</i> (See NOTE 1.)	50 \pm 20 %
<i>1.5 to 2.0 \times Minimum countable particle diameter of the counter</i>	100 \pm 10 %

NOTE 1: For a CNC, the minimum countable diameter shall be defined as a diameter having counting efficiency of 50 \pm 20%.

6.3.2 *False Count* — Equal to or less than 1 in 5 minutes.

6.3.3 *Acceptable Error of Flow Rate* — \pm 5%

6.4 *Sampler* — A system used to perform partial sampling of the gas stream exiting the test system, that consists of a diffuser equipped with a pressure reducing device in which gradual expansion to atmospheric pressure occurs and a sample probe. It is desirable that the sampler is designed to obtain isokinetic sampling. The average velocity of gas flowing through the sample probe should approximate the average velocity in the external tubing in which the sample probe is inserted. The sample probe overlap with the external tube must be of appropriate length, and it must be confirmed before use that the surrounding atmospheric gas is not drawn in the sample.

7 Test Condition

7.1 *Test Gas* — Liquid Nitrogen (LN₂) or cleaner Nitrogen (N₂) or Argon (Ar).

7.2 *Temperature* — Room temperature (between 18 and 26° Q

7.3 *Environment* — ISO/FDIS 14644-1 class 5 or better.

8 Preparation of Apparatus

8.1 Set up apparatus and the gas supply system as shown in Figure 1.

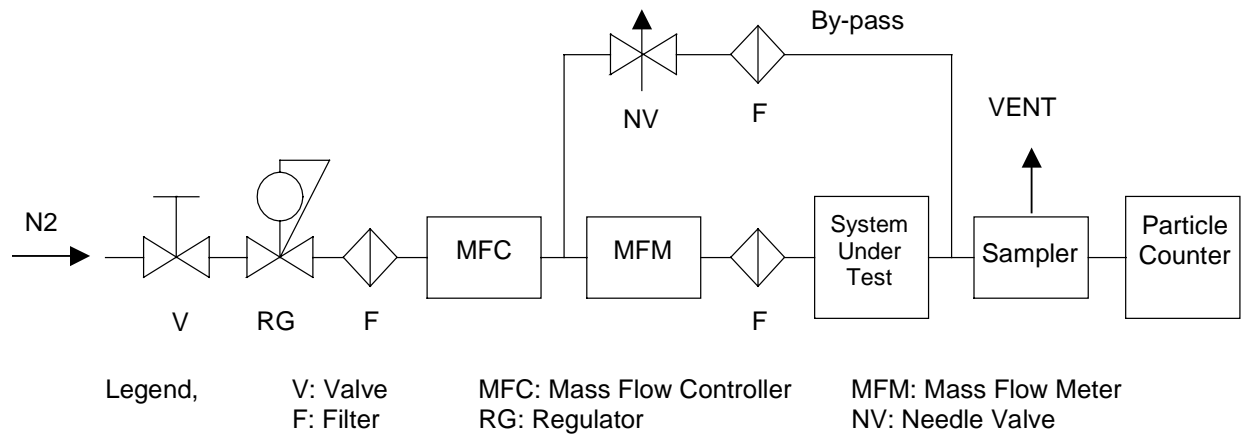


Figure 1
Test Set Up Basic Flow Diagram

8.2 If the test flow rate is equal to or less than the sample flow rate required by the particle counter, insert a filter in place of the sampler to fulfill the sample flow rate. (See Figure 2.)

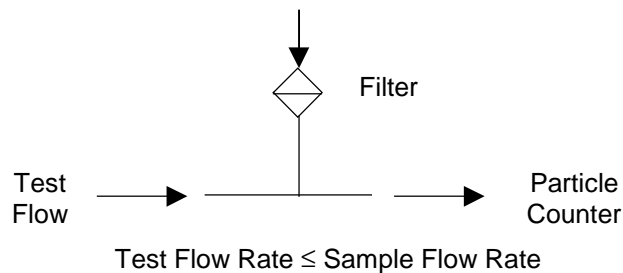


Figure 2
Full Sampling

8.3 If the test flow rate is higher than the sample flow rate required by the particle counter, use the sampler for partial sampling. (See Figure 3.) To establish the test flow rate and obtain an isokinetic condition, it is required to adjust the MFC in the test set up and the NV on the by-pass line. The by-pass line is required to have a capability to handle the maximum test flow when the valves in the system under test is closed position during the pulse test.

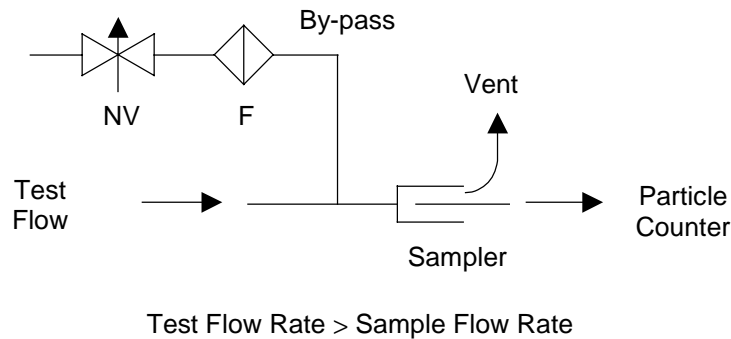


Figure 3
Partial Sampling

9 Procedure

9.1 A typical gas delivery system configuration is shown in Figure 4. The following test procedure shall be interpreted to apply to the actual test system and recorded by the operator.

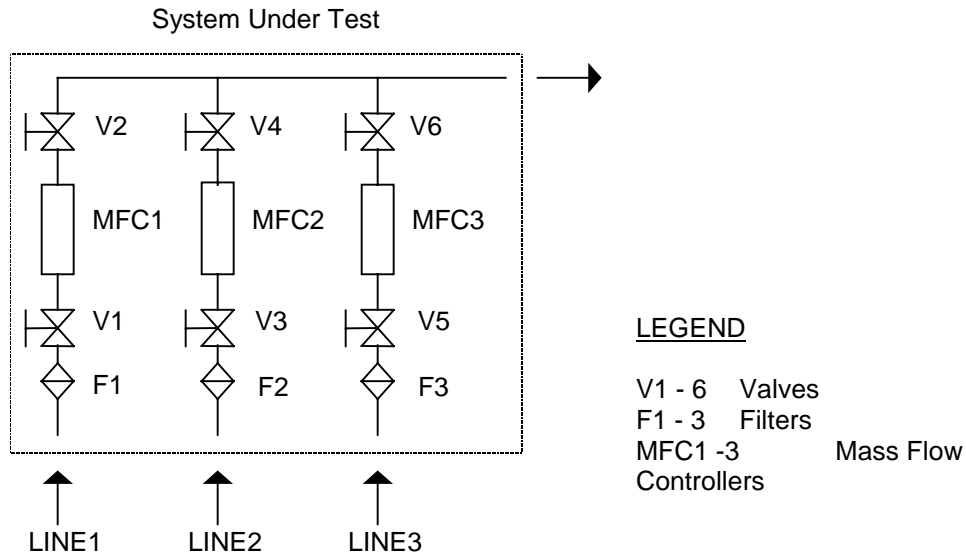


Figure 4
Typical Gas Delivery System Configuration

9.2 Determination of Background Counts

9.2.1 For test set up, insert a spool piece in place of the system under test (i.e. the gas delivery system) as in Figure 1.

9.2.2 Establish the test flow rate as 1.5 times of the design flow rate and supply N_2 (or Ar). In this case the design flow rate is determined as the flow rate of the line which has maximum flow rate in the system under test. Purge the test set up for more than 10 minutes.

9.2.3 Confirm the particle count is not more than 1 during 10 minutes after the purge. If the total volume of the test flow is less than 28.3 liters, extend the sampling time until it achieves more than 28.3 liters. If the particle count is more than 1, repeat Sections 9.2.2 and 9.2.3.

9.2.4 Replace the spool piece with the test system.

9.3 Static Flow Test

9.3.1 Close all valves in the line 2 and 3, set the MFCs as full scale or purge mode if available and fully open the valves in the line 1.

9.3.2 Supply N_2 (or Ar) to the line 1 with 1.5 times of the design flow rate of the line 1 and purge the test set up for more than 10 minutes. In many case, this will require the activation of the valve override on any MFCs on the test line. It is also required to verify that

all components in the system can withstand 1.5 times the design flow rate.

9.3.3 Count particles for more than 10 minutes. If the total volume of the test flow is less than 28.3 liters, extend the sampling time until it achieves more than 28.3 liters.

9.3.4 Repeat Sections 9.3.2 and 9.3.3 per all lines.

9.4 Pulse Flow Test

9.4.1 Close all valves in the line 2 and 3, set the MFCs as full scale or purge mode if available and fully open the valves in the line 1.

9.4.2 Supply N_2 (or Ar) to the line 1 with 1.5 times of the design flow rate of the line 1 and purge the test set up for more than 10 minutes.

9.4.3 Actuate V1 at the rate of 30 cycles/minute for 10 minutes if V1 is pneumatically operated valve. For manually operated valve, actuate the valve at 4 cycles/minute for 10 minutes. Each cycle consists of on and off actuation of the valve. The on and off cycles should be of equal duration. If the total volume of the test flow is less than 28.3 liters, extend the sampling time until it achieves more than 28.3 liters. Count particles during the period.

9.4.4 Repeat Section 9.4.3 for V2.

9.4.5 Repeat Sections 9.4.1 to 9.4.4 per all lines.

NOTE 2: For the pulse test, the total volume of the test flow shall be half of the test flow rate multiplied by the sampling time.

10.3 *Report Format* — Sample report formats are given in Appendixes 1 and 2.

10 Calculation and Report

10.1 The following test conditions are to be included in the test report:

- counter type (LPC or CNC)
- manufacturer of counter
- counter model (manufacturer's model # or part #)
- sample flow
- particle diameter
- sampler diameter
- sample probe diameter
- test gas
- background count
- valve operation interval (for the pulse flow test)
- flow diagram of the system under test

10.2 *Calculation of Data* — The test result is reported in number of particles in liters denoted by floating point representation with two significant digits (ex. $2.3 \times 10^{-2}/L$). The method of calculation is shown in Section 10.2.1.

10.2.1 The following formula is used to calculate the particles in a liter:

$$PARTICLES = \frac{N}{V}$$

where N: Total particle counts in 10 minutes or in the extended period if the total volume of the test flow is less than 28.3 liters.

V: Total sampling volume in liter in 10 minutes (mass flow meter read value) or in the extended period if the total volume of the test flow is less than 28.3 liters.

If the sample flow rate is equal to or more than the test flow rate, V is equal to the total volume of the test flow in liter in the sampling time.

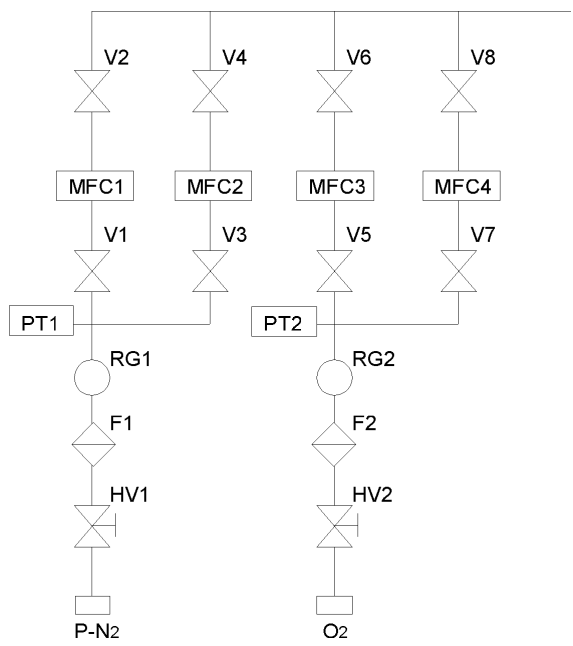
If the sample flow rate is less than the test flow rate, V is equal to the total volume of the sample flow in liter in the sampling time.

APPENDIX 1

SAMPLE REPORT FORMAT FOR STATIC FLOW PARTICLE TEST

NOTE: This appendix is being balloted as an official part of SEMI F70 by full letter ballot procedures, but the recommendation in this appendix are optional and are not required to conform to this standard.

STATIC FLOW PARTICLE TEST REPORT

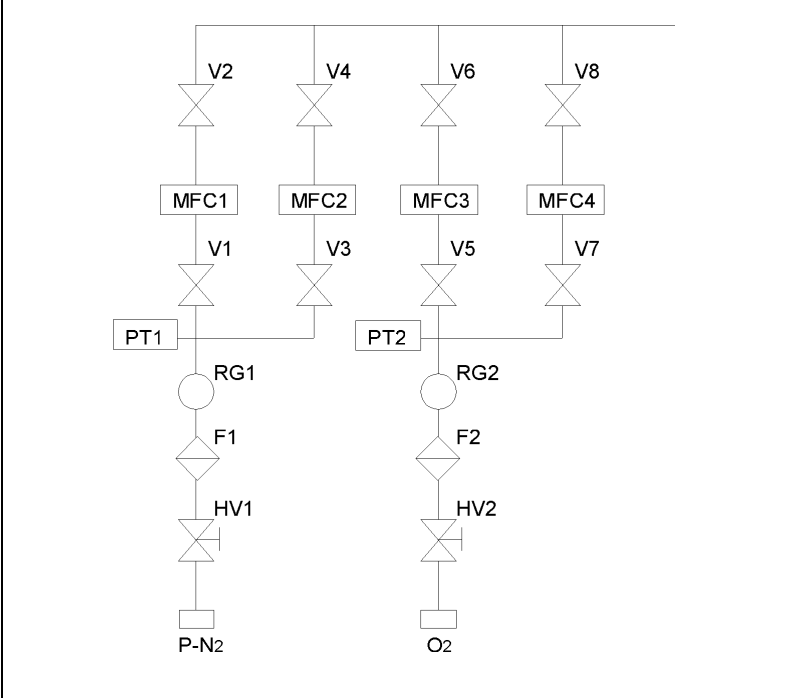
Line	Design Flow (l/min)	Test Flow (l/min)	Sampling Method	Total sampling volume (l)	Total particle count	Particle Count (particle/l)
P-N ₂	200	300	Partial Sampling	283	3	1.1×10^{-2}
O ₂	10	15	Full Sampling	150	2	1.3×10^{-2}
Flow Diagram of System Under Test				Test Condition		
				Counter Type: Manufacturer of the Counter Counter Model Sample Flow: Particle Diameter: Sampler Diameter: Sample Probe Diameter: Test Gas Background Count:		LPC A B 28.3 l/min $\geq 0.1 \mu\text{m}$ 30.0 mm 7.53 mm LN ₂ 0

APPENDIX 2

SAMPLE REPORT FORMAT FOR PULSE FLOW PARTICLE TEST

NOTE: This appendix is being balloted as an official part of SEMI F70 by full letter ballot procedures, but the recommendation in this appendix are optional and are not required to conform to this standard.

PULSE FLOW PARTICLE TEST REPORT

Line	Design Flow (l/min)	Test Flow (l/min)	Sampling Method	Total Sampling Volume (l)	Total Particle Count	Drive Valve #	Particle Count (particle/l)
P-N ₂	200	300	Partial Sampling	141.5	4	V1	1.4×10^{-2}
					3	V2	1.1×10^{-2}
					3	V3	1.1×10^{-2}
					4	V4	1.4×10^{-2}
O ₂	0.5	0.75	Full Sampling	28.5 (See NOTE 1.)	1	V5	3.5×10^{-2}
					1	V6	3.5×10^{-2}
					0	V7	0
					1	V8	3.5×10^{-2}
Flow Diagram of System Under Test					Test Condition		
 <p>The diagram illustrates the pulse flow particle test system. It features two parallel gas lines: P-N₂ and O₂. Each line starts with a pressure transducer (PT1, PT2), followed by a regulator (RG1, RG2), a flowmeter (F1, F2), and a high valve (HV1, HV2). The flow then passes through a series of four valves (V1-V4 for P-N₂, V5-V8 for O₂) and four mass flow controllers (MFC1-MFC4) before reaching the sampling points. The valves are controlled by a sequence of drive valves (V1-V8) as detailed in the table above.</p>					<p>Counter Type: LPC</p> <p>Manufacturer of counter: A</p> <p>Counter Model: B</p> <p>Sample Flow: 28.3 l/min</p> <p>Particle Diameter: $\geq 0.1 \mu\text{m}$</p> <p>Sampler Diameter: 30.0 mm</p> <p>Sample Probe Diameter: 7.53 mm</p> <p>Test gas: LN₂</p> <p>Background Count: 0</p> <p>Valve Operation Interval: 30 cycle/minute</p>		

NOTE 1. The test period for O₂ line was extended to 76 minutes in order that the total volume of the test flow achieves more than 28.3 liters.



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SEMI F71-1102

TEST METHOD FOR TEMPERATURE CYCLE OF GAS DELIVERY SYSTEM

This guideline was technically approved by the Global Facilities Committee and is the direct responsibility of the Japan Facilities Committee. Current edition approved by the Japan Regional Standards Committee on July 19, 2002. Initially available at www.semi.org September 2002; to be published November 2002.

1 Purpose

1.1 The purpose of this document is to provide a standardized methodology and procedure for the leakage performance of gas delivery systems temperature cycling.

2 Scope

2.1 This test method applies to gas delivery systems installed in semiconductor manufacturing equipment.

2.2 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 Referenced Standards

NOTE 1: As listed or revised, all documents cited shall be the latest publications of adopted standards.

3.1 SEMI Standards

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

4 Terminology

4.1 Definitions

4.1.1 *gas delivery system* — A system installed in semiconductor manufacturing equipment to deliver process and carrier gases to reactors, which typically consists of tubing, fittings, valves, filters, mass flow controllers and regulators.

4.1.2 *Helium leak test* — Testing shall be conducted per procedure outlined of inboard leak test in SEMI F1.

4.1.3 *T-max* — The maximum set temperature of the temperature cycle test.

4.1.4 *T-min* — The minimum set temperature of the temperature cycle test.

5 Apparatus

5.1 *helium leak detector* — The detector has appropriate detection sensitivity according to SEMI F1.

5.2 *constant temperature oven* — The constant temperature oven should have the capacity to accommodate the test configuration and maintain the elevated T-max temperature to $\pm 5^{\circ}\text{C}$.

5.3 *freezer* — The freezer should have the capacity to accommodate the test configuration and maintain the T-min temperature to $\pm 5^{\circ}\text{C}$.

5.4 *thermocouple* — The thermocouple should make contact with the sample and should take measures of a noise.

6 Test Specimen and Condition

6.1 The temperature cycling test should be performed using a typical gas delivery system configuration. An example is shown in Figure 1.

6.1.1 The influence of heat expansion is not easily measured with a single component. Therefore, use a gas panel having a minimum of three lines as an assembled sample. An example is shown in Figure 1.

6.2 A set temperature (T-max, T-min) at the temperature cycle must be within the range of the rated temperature of components used in the gas panel.

6.3 The sealing surface used in the gas delivery system shall not have any scratches or other defects which are visible to non-magnified normal vision.

6.4 Install the thermocouple in approximately the center of the gas panel to be tested.

6.5 The user can arbitrarily decide heating and cooling temperatures in temperature cycle examination according to the purpose of use.

6.6 The helium leak test shall be performed only at room temperature ($22 \pm 4^{\circ}\text{C}$), not during heating or cooling.

7 Procedure

7.1 A typical gas delivery system configuration is shown in Figure 1. The following test procedure shall be interpreted to apply to the actual test target and deviations from the procedure in this document shall be recorded along with the test results. (The condition of heated T-max and cooling T-min was taken up here as an example.)

7.2 Perform the helium leak test and confirm whether there is leakage in the gas panel before starting temperature cycle examination. Connect all flow path in the gas panel to helium leak detector and plug all open end connections before the helium leak test is executed. Abort testing if leakage is detected and record leaking points and leak rate.

7.3 Put the test sample in the constant temperature oven and apply heat until the test sample reaches T-max. Maintain the temperature for 10 ± 1 minutes.

7.4 Remove the sample from the constant temperature oven and cool down to the room temperature and perform helium leak test.

7.5 Put the test sample in the freezer and cool until the test sample reaches T-min. Maintain the temperature for 10 ± 1 minutes.

7.6 Remove the sample from the freezer and equilibrate to the room temperature and perform helium leak test.

7.7 Repeat Section 7.2 through 7.6 for 5 cycles. The temperature cycles should be recorded and plotted as shown in Figure 2.

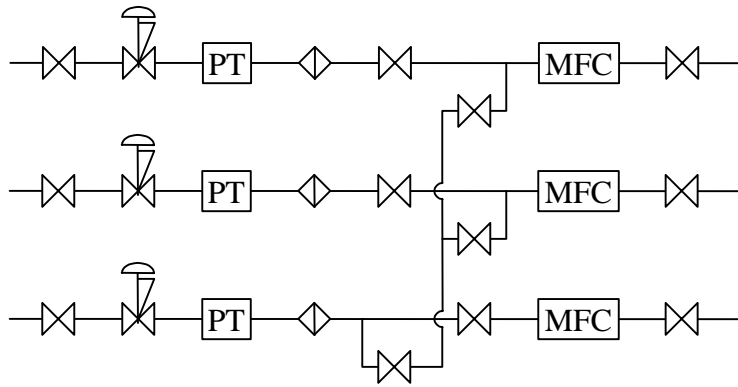


Figure 1
Typical Gas Delivery System Configuration

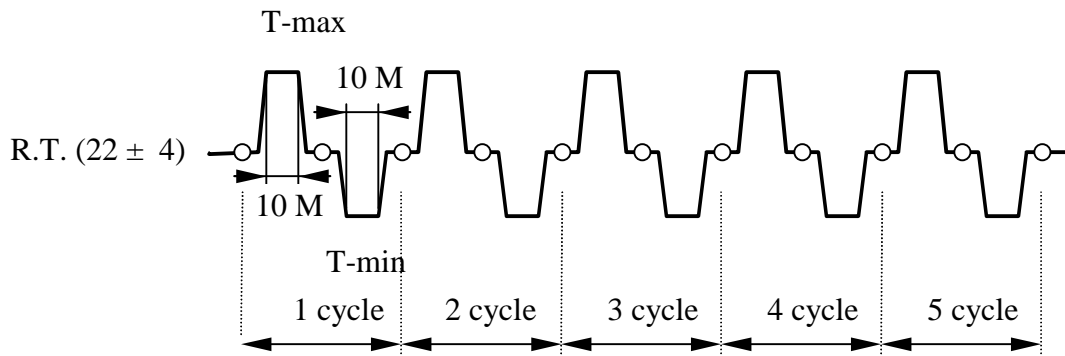


Figure 2
Temperature Cycle Chart

8 Reporting Results

8.1 The report shall include the following:

- Date and time of test,
- Operator,
- Leak point and leak rate,
- Helium leak detector maker and model number,
- Flow diagram of the test gas panel and layout drawing,
- Schematic of the test system,
- Temperature conditions of the temperature cycle test, and
- Any deviations from the test procedure in section 7.

A sample report format is given in Appendix 1.

APPENDIX 1 SAMPLE REPORT FORM

NOTE: The material in this appendix is an official part of SEMI [insert designation, without publication date (month-year) code] and was approved by full letter ballot procedures on July 19, 2002

A1-1 Sample Report Form

Test date:

Test operator:

Helium leak detector maker and model number:

Table A1-1 TEMPERATURE CYCLE TEST REPORT

<i>Number of Heat Cycle</i>		<i>Leak Point and Leak Rate</i> <i>(Pa × m³/sec)</i>	<i>Background</i> <i>(Pa × m³/sec)</i>
Initial		N.D.	3.6×10^{-11}
1	after heating (T-max)	N.D.	3.1×10^{-11}
	after cooling (T-min)	N.D.	4.1×10^{-11}
2	after heating (T-max)	N.D.	2.6×10^{-11}
	after cooling (T-min)	N.D.	3.3×10^{-11}
3	after heating (T-max)	N.D.	4.3×10^{-11}
	after cooling (T-min)	N.D.	3.0×10^{-11}
4	after heating (T-max)	N.D.	2.1×10^{-11}
	after cooling (T-min)	N.D.	1.6×10^{-11}
5	after heating (T-max)	N.D.	2.5×10^{-11}
	after cooling (T-min)	N.D.	3.0×10^{-11}

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SEMI F72-1102

TEST METHOD FOR AUGER ELECTRON SPECTROSCOPY (AES)

EVALUATION OF OXIDE LAYER OF WETTED SURFACES OF PASSIVATED 316L STAINLESS STEEL COMPONENTS

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 July 21, 2002. Initially available at www.semi.org October 2002; to be published November 2002.

1 Purpose

1.1 The purpose of this document is to define a test method to characterize the surface composition of passivated 316L stainless steel components being considered for installation into a high-purity gas distribution system. This test method is intended to be applied to the wetted surfaces of stainless steel tubing, fittings, valves, and other components as a measure of the effectiveness of passivation.

1.2 The objective of this method is to describe a general set of instrument parameters and conditions that will achieve reproducible measurements within the chromium-enriched passive oxide layer.

2 Scope

2.1 This document describes a test method to characterize the composition and thickness of the chromium-enriched oxide layer of stainless steel surfaces and to detect surface contamination in tubing, fittings, valves and other components. The procedure involves detection and measurement of the surface elemental composition by Auger Electron Spectroscopy (AES). This procedure also describes the test method for a depth compositional profile of Cr, Fe, Ni, O, and C from the as-received surface, through the oxide layers, and extending into the base metal. This measurement provides oxide thickness and chromium enrichment information throughout the passivated region.

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

3 Limitations

3.1 This test method is intended to be used by AES analysts familiar with the instrumentation and technique. The AES instrument must be calibrated and maintained to pertinent manufacturer's specifications. The method is not intended to preclude the use of any

particular brand or model of surface analysis equipment. While most of the test methodology has been developed using specific instrumentation, this method can be adapted to most Auger surface analytical instrumentation.

3.2 Quantification of the elemental compositions is performed with handbook values of the relative elemental sensitivity factors. These sensitivity factors do not allow for differences due to the chemical environment of the elements, and are thus not accurate in this instance in which the chemical environment changes from the passive oxide layer to the metal alloy. In addition, quantification is affected by the choice of instruments and instrument parameters. For these reasons the results of this test method may not be reproducible between different instruments and operators. Use of the results of this test method should be restricted to process development and comparison to an historical database of AES data from the same source.

3.3 The effects of the depth of analysis of the technique and surface contamination affect the results of this test method. These are discussed in the attached appendix. Surface roughness, non-planarity of the surface, and differential sputtering rates for the different chemical species also cause measurement uncertainties in this test method.

3.4 The results of this test method have not been demonstrated to affect performance of stainless steel components in high purity gas distribution systems for semiconductor manufacturing.

4 Referenced Standards

4.1 SEMI Standard

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

4.2 ASTM¹ Standards

ASTM E 673 — Standard Terminology Relating to Surface Analysis

ASTM E 1078 — Standard Guide for Specimen Handling in Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy

ASTM E 1127 — Standard Guide for Depth Profiling in Auger Electron Spectroscopy

NOTE 1: Unless otherwise indicated, all documents cited shall be the latest published versions

5 Terminology

5.1 Terminology is per ASTM E 673 unless otherwise specified.

5.2 Abbreviations and Acronyms

5.2.1 AES — Auger Electron Spectroscopy

5.3 Definitions

5.3.1 *passivation* — the chemical treatment of a stainless steel surface with a mild oxidant for the purpose of enhancing the corrosion resistant surface film.

5.3.2 *sampling volume* — the volume in the sample from which Auger electrons are detected. The electron beam spot size or the scan area, and the acceptance angle of the electron analyzer determine the lateral dimensions. A length of three times the Auger electron mean free path is considered the maximum depth sensitivity. Sampling volume is dependent on the sample material and TOA.

5.3.3 *take-off angle (TOA)* — the angle that the collection lens forms with the sample plane.

6 Summary of Method

6.1 Data Acquisition

6.1.1 Acquire initial elemental survey and calculate elemental composition of “as received” wetted surface.

6.1.2 Acquire a compositional depth profile by ion etching to determine the relative abundance of C, O, Cr, Fe and Ni. Additional elements may be included as desired (i.e., molybdenum, silicon and nitrogen). The thickness of the passive oxide layer and carbon is also determined from the depth profile.

6.2 *Reporting* — Data is provided consisting of:

6.2.1 An initial survey spectrum extending from approximately 0 to 2000 eV.

6.2.2 A compositional depth profile plot including C, O, Cr, Fe and Ni as a function of sputtering time.

6.2.3 A table of the as-received surface elemental composition calculated from the initial survey spectrum.

6.2.4 A table of the oxide thickness, the carbon thickness, and the maximum of the Chromium to Iron ratio calculated from the depth profile.

7 Possible Interferences

7.1 *Cr and O* — Carefully select windows for oxygen and chromium to minimize interference. Monitor individual windows after profile is complete to evaluate effects. Some instruments may have enhanced ability to compensate for overlaps.

8 Apparatus

8.1 *Instrumentation* — Any AES instrument equipped with an ion gun may be used, whether it etches and measures simultaneously or in alternating fashion. An instrument that analyzes and etches in alternating fashion should be evaluated to assure that no significant oxygen level redeposits onto the sample surface between etching intervals. The electron analyzer may be of either the hemispherical or cylindrical mirror analyzer (CMA) type. The electron energy analyzer shall be of high enough energy resolution to permit adequate separation of the Chromium KLL and Oxygen KLL Auger peaks.

8.2 Instruments with geometries significantly different from one another may provide analysis from different sampling volumes. The incident electron beam energy, the angle of the incident electron beam to the sample plane, and the take-off angle must be recorded.

9 Reagents and Materials

9.1 *Instrument Calibration Materials* — Refer to instrument manufacturer recommendations or ASTM E 1127 for standard materials.

10 Safety Precautions

10.1 This test method does not purport to address the safety considerations associated with use of high voltage, vacuum, and electron producing equipment. The method assumes an AES analyst with adequate skill level as well as knowledge of instrumentation and associated safety precautions.

1 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

11 Test Specimens

11.1 Specimens are to be sectioned to appropriate size for the particular AES instrument using a clean, dry hacksaw or dry low speed bandsaw. Any sample preparation shall avoid introducing contamination onto the surface to be measured. Clean noncontaminating gloves and tweezers should be used to handle samples, avoiding contact with the area of interest. In addition, preparation must avoid excessive heating of the sample; i.e., the surface temperature shall not exceed 50°C, to avoid oxide growth or change in surface composition.

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

11.3 If sample preparation is done by other than the manufacturer, the sample(s) may be cleaned in DI water and dried promptly. If the sample(s) are not to be analyzed immediately they should be packaged by wrapping in clean metal foil or sealing in cleanroom quality nylon bags.

11.4 If sample preparation is done by other than the manufacturer this shall be stated in the report narrative and the analytical results are not to be interpreted as indicative of the manufacturer's quality of cleaning and packaging procedures. A note to this effect shall be included in all tables of reported results of the composition of the surface.

11.5 After preparation, samples should be analyzed promptly, with allowance for shipping times and queuing time at the analyst.

12 Preparation of Apparatus

12.1 Instruments shall be routinely tested in accordance with manufacturer recommendations to assure proper performance. The instrument vacuum shall be 1.0×10^{-7} Torr or better during the analysis.

13 Calibration and Standardization

13.1 Instrument calibration for etch rates and sensitivity factors shall be performed in accordance with instrument manufacturer recommendations or other established method.

14 Procedure

14.1 The sample is to be mounted in accordance with manufacturer's recommendations and in a manner consistent with ultra-high vacuum surface analytical procedures. Some of these practices are detailed in ASTM E 1078. The area to be analyzed should be

mounted parallel to the sample holder surface so that the TOA is known.

14.2 Place the sample in the AES introduction chamber for pumpdown. Transfer to the analytical chamber at the manufacturer's recommended base pressure.

14.3 Align the sample with respect to the electron beam and analyzer so that optimum count rate from the desired analytical location is obtained. The surface area to be analyzed should be free of sample preparation debris, visible particles and large defect features, if possible.

14.4 A large beam size or scan area should be used to attempt measurement of a representative surface. Elemental survey data (approximately 0-2000 eV) are to be measured from the sample surface to determine the elements present and their approximate surface abundances. A signal-to-noise ratio of 2 is usually adequate to ensure detection of elements present at one atomic percent or greater levels. A typical survey spectrum is shown in Figure 1.

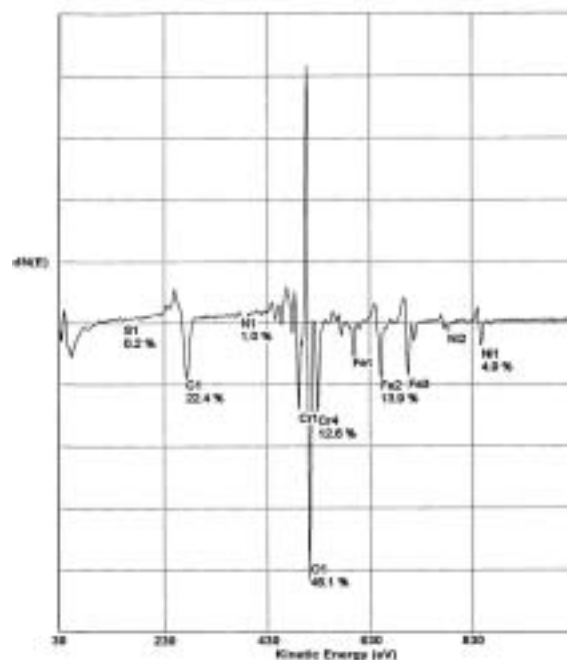


Figure 1
Auger Spectrum of Stainless Steel Surface

14.5 Acquire a depth composition profile to determine the relative abundances of Cr, Fe, Ni, O, and C. The profile may be acquired in a simultaneous or an alternating etch/data acquisition mode. Measurement of each element shall be made at a frequency of at least one data point every 5 Å within the first 50 Å and one data point every 10 Å from 50 Å to the profile end. An appropriate scan acquisition time is one that optimizes

spectral resolution and signal/noise, and that has at least ten data points in each spectral window. The depth profile should be continued into the depth of the sample until the Fe, Cr and Ni levels are approximately constant.

14.6 Depth profile data are presented as signal intensity of each element with etch time (i.e., depth), and as atomic concentration with etch time. Depths may be estimated from etch times by determination of the etch rate on a known thickness of standard material. A typical AES profile is shown in Figure 2.

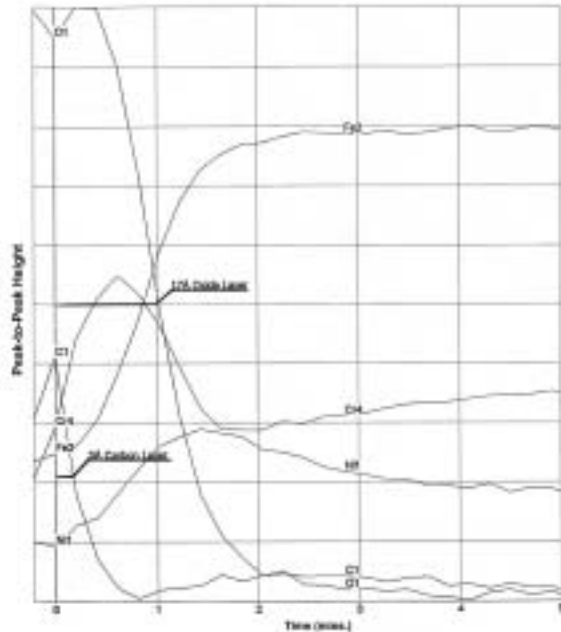


Figure 2
Auger Depth Profile of Stainless Steel

15 Calculations and Interpretation of Results

15.1 Most manufacturers supply software for calculation of the approximate elemental composition from the survey spectrum and the depth profile data. The elemental composition should be estimated using sensitivity factors appropriate to the instrument, each element, and resolution settings for each measurement. AES spectra are considered only semiquantitative, due to instrument geometry and the chemical form dependency of each element's sensitivity factor. Evaluations of elemental composition should therefore be limited to similar samples analyzed on the same instrument.

15.2 The signal intensity with etch time graph may be used to calculate oxide thickness, defined as the point at which the oxygen signal decreases to half its maximum value. The thickness of surface carbon contamination is calculated in the same way from the carbon signal.

15.3 Relative abundances of profiled elements are recorded on the atomic concentration with etch time graph.

16 Reporting Results

16.1 A tabular summary of estimated surface elemental composition is to be supplied with its associated elemental survey spectrum.

16.2 Tabular summaries of oxide thickness, thickness of the region in which Cr concentration exceeds Fe concentration, thickness of a surface Fe enrichment (if present), and carbon thickness are to be supplied with associated depth profile graphs.

16.3 Optionally, parameters such as maximum Cr/Fe ratio may be reported. However, the value of the maximum Cr/Fe ratio is subject to instrument geometry and sensitivity factor variations, and should not be used for primary evaluation purposes. Comparative evaluations should only be made from data collected by the same instrument with an identical protocol.

16.4 Data acquisition parameters, including instrument geometry, electron beam size or scan area, and other pertinent settings are to be supplied. The manufacturer and model number of instrument used is to be reported. Analyst identity and analysis date are also required information. Each table and graph must be clearly labeled with sample identification.

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APPENDIX 1

DISCUSSION OF EFFECTS OF DEPTH OF ANALYSIS ON DEPTH PROFILE ANALYSIS BY AUGER ELECTRON SPECTROSCOPY FOR SURFACE CHEMISTRY ANALYSIS OF PASSIVATED STAINLESS STEEL

NOTE: The material in this appendix is an official part of SEMI F72 and was approved by full letter ballot procedures. This appendix was derived from presentations made to the Stainless Steel and Surface Analysis Workshop².

A1-1

A1-1.1 The purpose of this appendix is to describe the Auger Electron Spectroscopy (AES) technique and explain the interpretation of the depth profile analysis with respect to the structure and composition of the passive oxide layer on stainless steel. The effect of the depth of analysis on the measured chromium to iron ratio, the oxide thickness, and the depth of enrichment is discussed. The discussion also applies to depth profile analyses performed by ESCA (Electron Spectroscopy for Chemical Analysis, sometimes referred to as X-Ray Photoelectron Spectroscopy, or XPS).

A1-1.2 *Auger* — The mechanism of Auger electron generation is illustrated in Figure A1-1. The energy of the Auger electron is determined by the energies of the electron energy levels participating in the generation process and are unique for each chemical element. Thus the elements present in the analyzed region may be determined by analysis of the energies of emitted Auger electrons.

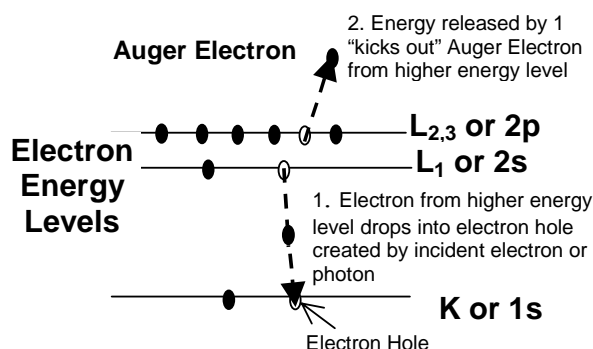


Figure A1-1
Schematic Representation of Auger Electron Generation Process

A1-1.3 The Auger technique makes use of an electron beam for primary excitation, typically of energy in the range 2 to 5 keV. The electron beam may be defocused to analyze a relatively large area of the surface, of the order of micrometers, or focussed and rastered to “map” the distribution of elements on the surface. An electron energy analyzer is focused at the same point as the incident electron beam, and the Auger electrons that escape the surface are detected and analyzed. The analysis is commonly presented as the derivative of the electron signal intensity N as a function of energy E , $dN(E)/dE$, of the spectrum to enhance the visibility of the small Auger electron peaks relative to the background. An example of an Auger electron spectroscopy spectrum of a stainless steel surface is shown in Figure 1 of this test method.

A1-2 Illuminated Volume and Surface Sensitivity

A1-2.1 The incident electron beam penetrates well below the surface. Auger electrons are generated all through this illuminated volume. The Inelastic Mean Free Path (IMFP) of an electron in a solid is the mean distance through the solid that an electron can travel before losing some of its energy (suffering an inelastic interaction). The IMFP is a function of the electron’s energy and the matrix through which it is traveling. Electrons with an energy typical of Auger and ESCA electrons, approximately 1 keV, have an IMFP of about 10 angstroms. That means that a 1 keV electron will travel, on average, 10 angstroms before interacting with the matrix and giving up some of its energy. In other words it will be scattered to lower energy and will not be part of the photoelectron or Auger electron line for its element. The IMFP is a statistical parameter and an electron may travel several IMFPs before having a collision. The “several IMFPs” is called the escape depth of the electron, or the depth of analysis, as shown

² Stainless Steel and Surface Analysis Workshop, March, 1999; *Interpretation of Depth Profile Analysis Data*, Dave Harris, Charles Evans and Associates.

in Figure A1-2. Although Auger and ESCA are considered surface analytical tools, their depth of analysis is on the order of 50 angstroms, or about one to three times the depth of the oxide film found on a typical passivated stainless steel surface.

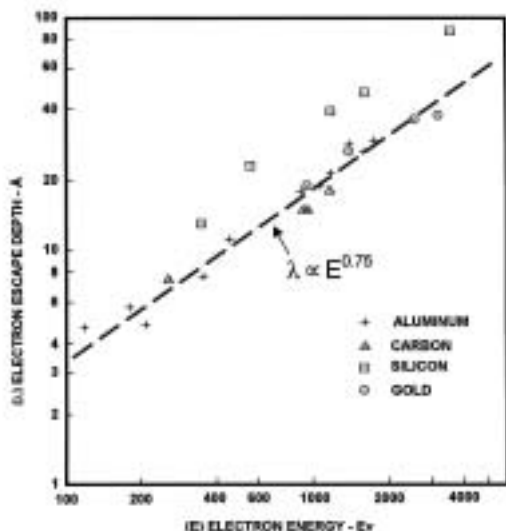


Figure A1-2
The Electron Escape Depth of Low Energy
Electrons in Some Metals

A1-3 Depth of Analysis Function

A1-3.1 There has been some discussion with respect to the “Depth of Analysis Function”. There is one school that believes that it is 5 IMFPs and another that uses 3 IMFPs. These two functions will be referred to as 5λ and 3λ respectively. It will be seen that it makes little difference which function is used even though the depth of the oxide films is of the order of the functions. These functions are seen in Figure A1-3, which shows the proportion of the total signal generated from each depth below the surface. The Y-axis is in decimal and the X-axis is in angstroms from the surface. The first point of the 5λ curve is 0.16 at the 1st angstrom level. That means that 16% of the signal comes from the 1st angstrom, 14% from the 2nd angstrom level and so on down to 1% from the 25th angstrom level. Thus elements that do not appear on the surface or in the first 24 angstroms in from the surface, but do exist at the 25 angstrom level will be detected.

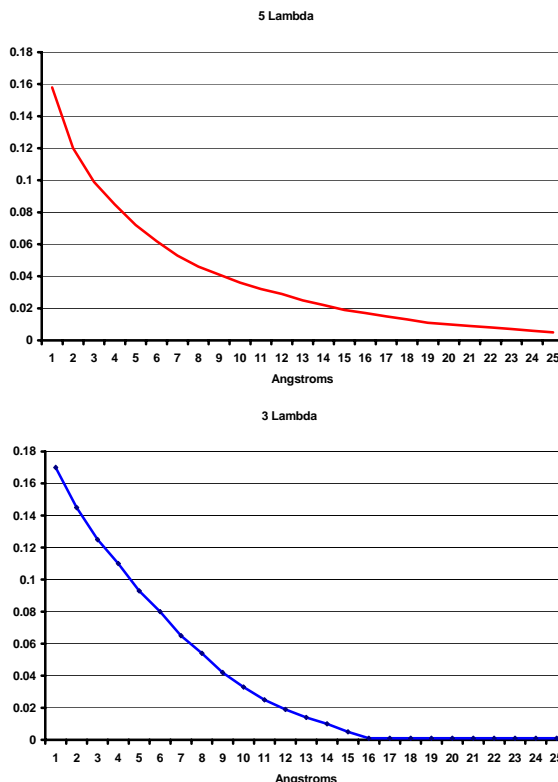


Figure A1-3
These Two Graphs Represent the “Depth of
Analysis” That Will be Seen When Analyzing 1 keV
Electrons. The Top Curve is the 5λ Curve and
Bottom the 3λ .

A1-4 Modeling

A1-4.1 Figure A1-4 is the composition profile of a “perfect” oxide. It represents 25 angstroms of pure Cr_2O_3 on an atomically flat 316L stainless steel surface, with no surface or interfacial contamination present. Figures A1-5a and b are the theoretical depth profiles derived by assuming a depth of analysis of 5λ and 3λ respectively. The first factor to note is that Fe, Ni and Mo appear most or all of the way through the depth profile of the oxide due to the contribution of the metal substrate to the detected signal, the “depth of analysis” effect as introduced above. 1 keV electrons can escape from as deep as 25 angstroms. This must be appreciated when interpreting these depth profiles.

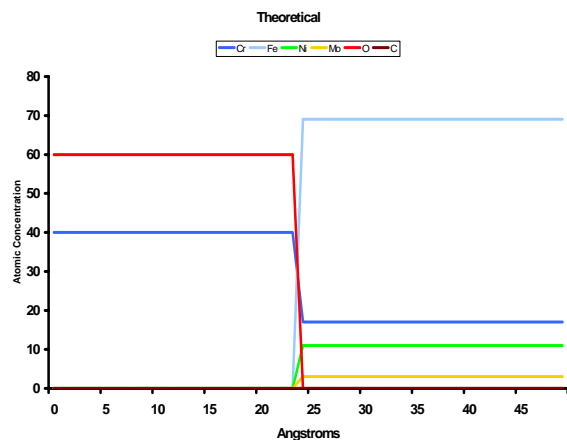


Figure A1-4
Composition Versus Depth of a Pure Cr_2O_3 Layer on Stainless Steel

A1-4.2 The second factor to notice is the thickness of the oxide. Historically, the FWHM (Full Width Half Maximum; the width of a peak measured at half its maximum height) of the oxygen profile has been used as the measure of oxide thickness. It is apparent in Figures A1-4 and A1-5 that this underestimates the oxide thickness by 15 to 20 %.

A1-4.3 It should also be noted that the Cr:Fe ratio measured from Figures A1-4 and A1-5 has its maximum value at the initial surface, decreasing as the depth profile progresses, whereas the actual Cr:Fe ratio in Figure A1-4 is infinite down to 25 angstroms. Most actual depth profiles exhibit a maximum of the Cr:Fe ratio at some depth below the initial surface, referred to as the depth of maximum enrichment. This is a consequence of variation in the actual composition of the oxide, possibly having a higher concentration of Fe near the surface, a phenomenon commonly termed a “detached iron oxide layer”, or of contamination on the surface, generally hydrocarbons. These are discussed in the next section

A1-5 Effects of Detached Iron Oxide Layer and Surface Contamination

A1-5.1 The model composition profile of Figure A1-6 shows 3 angstroms of pure Fe_2O_3 over 22 angstroms of pure Cr_2O_3 on 316L stainless steel. This is a model of a detached iron oxide layer. Figure A1-7 is the theoretical depth profile of this model derived assuming a depth of analysis of 5λ . Note that the initial Cr value in the profile is higher than the Fe value, even though the surface is pure Fe_2O_3 . This is a consequence of the depth of analysis detecting the Cr from levels beneath the surface. The Cr:Fe ratio for this model has its maximum value at 3 angstroms below the initial

surface. The theoretical depth profile using a depth of analysis of 3λ is very similar

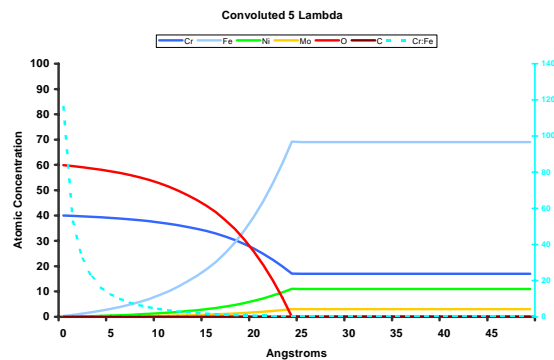


Figure A1-5a
Theoretical Depth Profile of Figure 5 Assuming 5λ Depth of Analysis

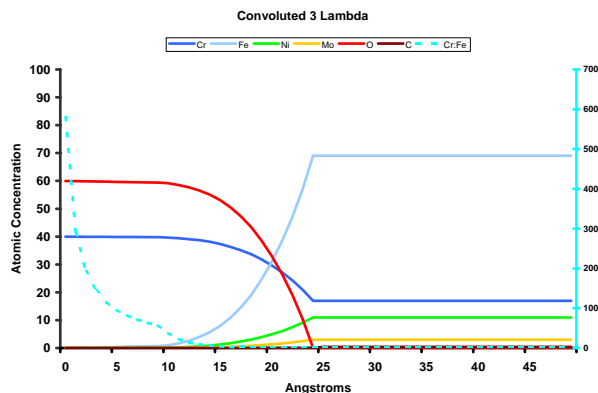


Figure A1-5b
Theoretical Depth Profile of Figure 5 Assuming 3λ Depth of Analysis

A1-6 Effects of Hydrocarbon Contamination

A1-6.1 The model composition profile of Figure A1-8 shows 3 angstroms of pure carbon over 22 angstroms of pure Cr_2O_3 on 316L stainless steel, representing an idealized model of the adsorbed hydrocarbon contamination generally found on stainless steel surfaces exposed to the atmosphere. Figure A1-9 is the theoretical depth profile of this model assuming a depth of analysis of 5λ . In this case the O, Cr and Fe atomic concentration values are reduced by the presence of the carbon until the depth profiling proceeds past the carbon, but they have the same relative values (ie: same Cr:Fe ratio) versus depth as derived in the uncontaminated model. The maximum of the Cr:Fe ratio is seen to be at the initial surface. Although the actual oxide thickness is less in this model, the FWHM measure of the oxide thickness from the depth profile is the same due to the presence of the carbon layer. Note

that the Oxygen concentration profile initially increases to a maximum, then decreases. This is typical of Oxygen concentration profiles seen on actual samples, which will generally have some adsorbed hydrocarbon contamination on the surface.

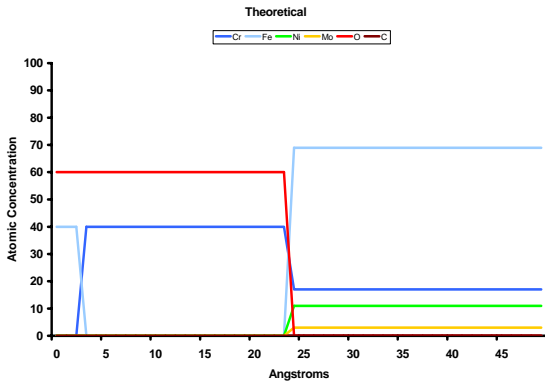


Figure A1-6
Composition Versus Depth of a Fe_2O_3 Layer Over a Cr_2O_3 Layer on Stainless Steel

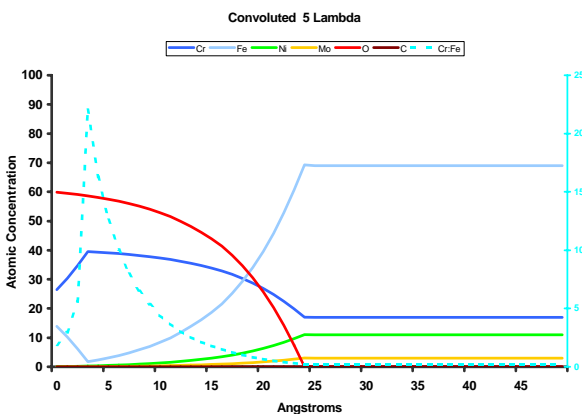


Figure A1-7
Theoretical Depth Profile of Figure A1-6 Assuming 5λ Depth of Analysis

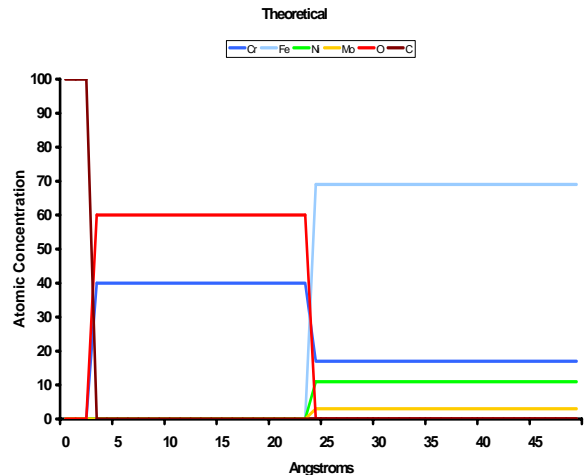


Figure A1-8
Composition Versus Depth of a Carbon Layer Over a Cr_2O_3 Layer on Stainless Steel

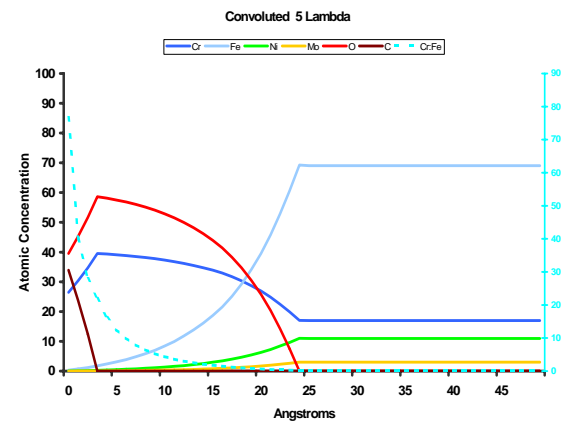


Figure A1-9
Theoretical Depth Profile of Figure A1-8 Assuming 5λ Depth of Analysis

A1-7 Effects of Hydrocarbon Contamination over Detached Iron Oxide Layer

A1-7.1 The model composition profile of Figure A1-10 shows a 3 angstrom carbon layer over 3 angstroms of pure Fe_2O_3 over 19 angstroms of pure Cr_2O_3 on 316L stainless steel. Figure A1-11 is the theoretical depth profile of this model assuming a depth of analysis of 5λ . This profile is seen to be similar to the depth profiles generally observed for passivated stainless steel. The Cr:Fe ratio maximum occurs below the initial surface, and the initial surface atomic concentrations of the elements of interest are diluted by the presence of the carbon layer on the surface. The oxide thickness, as measured by the FWHM technique, is affected by both

the presence of the carbon layer and the depth of analysis.

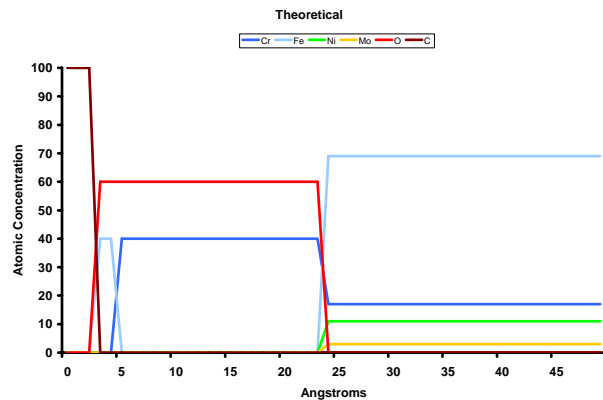


Figure A1-10
Composition Versus Depth of a Carbon Layer Over a Fe_2O_3 Layer Over a Cr_2O_3 Layer on Stainless Steel

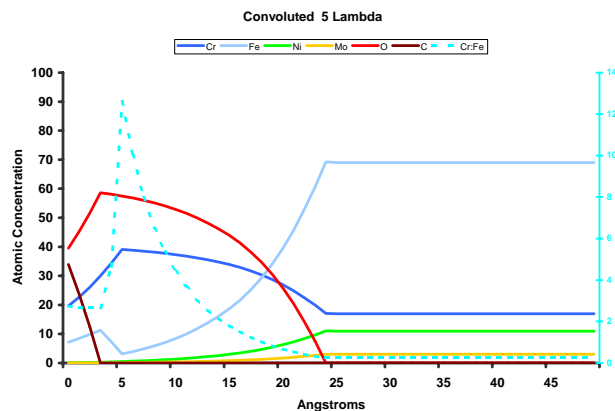


Figure A1-11
Theoretical Depth Profile of Figure 11 Assuming 5λ Depth of Analysis

A1-8

A1-8.1 It must be emphasized that these derived depth profiles are for models with perfect interfaces and perfect compositions instead of the compositional gradients observed in real systems. Additional measurement uncertainties result from roughness and non-planarity of the surface, and from differential sputtering rates for different chemical species during depth profiling.

A1-8.2 The depth profiles of real systems must be interpreted with an understanding of the effects described in this appendix and a realization that they are not ideal. The same considerations pertain to ESCA depth profile analysis.

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SEMI F73-1102

TEST METHOD FOR SCANNING ELECTRON MICROSCOPY (SEM)

EVALUATION OF WETTED SURFACE CONDITION OF STAINLESS STEEL COMPONENTS

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 August 29, 2002. Initially available at www.semi.org September 2002; to be published November 2002.

1 Purpose

1.1 This document defines a uniform procedure for testing the wetted surfaces of stainless steel components intended for installation into high purity gas distribution systems. This procedure characterizes the occurrence, frequency, and in some cases the identity of microscopic surface defects and contaminants that may appear on the wetted surfaces. It should be noted that there has been no direct correlation made between the results of this test method and contamination of process gases or product yields in processes served by high purity gas distribution systems. Application of this test method is intended to yield comparable and reproducible results among various users of this method for the purposes of qualification of components.

1.2 The objective of this method is to describe a general set of instrument parameters and conditions that will achieve precise and reproducible measurements of important parameters regarding the surface condition.

2 Scope

2.1 This procedure applies to the wetted surfaces in stainless steel tubing, fittings, valves, and other components to determine the effectiveness of surface finishing and cleaning processes. The technique describes counting of surface defects including pits, inclusions, inclusion stringers, scratches, residual process marks, grain boundaries and contamination on the wetted surfaces. However, any surface damage produced during sample preparation is to be excluded from such assessment.

2.2 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 This test method is primarily intended to determine the frequency of occurrence (surface density) and identity of microscopic surface defects, down to a size range of \geq approximately 0.1 micrometer, that may be

detrimental to high purity gas distribution systems. Such defects may not be identifiable by visual or magnified optical inspection, but concentrations of microscopic surface defects may cause visually apparent defects such as haze.

3.2 This test method requires sectioning of the specimen(s) used for test purposes and is therefore destructive.

3.3 The method must be applied to representative examples of process lots to determine quality of the processes employed and/or lots processed. As the areas examined and measured by this method are very small, the results may not be representative of all areas or all lots processed.

3.4 This test method may be subject to operator bias in selection of representative areas and definition of countable defects.

3.5 Detection of countable defects is affected by operator selection of SEM operating conditions and image recording conditions. This test method assumes that the operator is sufficiently proficient in operation of the SEM to minimize this limitation, per instructions in the procedures.

3.6 SEM imaging will reveal surface finish flaws, but may not show features that are well rounded by an electropolishing or other surface leveling process.

3.7 Energy dispersive X-ray spectroscopy (EDS) may be used in this test method to analyze surface contaminants at least approximately one micrometer in thickness and inclusions at least approximately one micrometer in size. EDS is not an appropriate technique for analysis of the oxide passive layer on the stainless steel surface, as EDS analyzes down to a depth of the order of 1 micrometer below the surface, and the oxide passive layer is only 0.001 to 0.01 micrometer deep.

4 Referenced Standards

4.1 ASTM Standards¹

ASTM E 7 — Standard Terminology Relating to Metallography

ASTM E 766 — Standard Practice for Calibrating the Magnification of a Scanning Electron Microscope

4.2 ANSI/IEEE Standard³

759 — Standard Test Procedures for Semiconductor X-ray Energy Spectrometers

NOTE 1: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 Abbreviations and Acronyms

5.1.1 *EDS* — Energy Dispersive X-ray spectroscopy, sometimes called EDX.

5.1.2 *FWHM* — abbreviation of Full Width Half Maximum; the width of an EDS peak measured at half its maximum height.

5.1.3 *SEM* — Scanning Electron Microscopy.

5.2 Definitions

5.2.1 *defect* — any surface feature that is either characteristic of the material, or a result of material processing or product fabrication, that is deemed capable of generating and/or trapping and releasing particles, or otherwise be detrimental to the contained fluids. Specific features that may be considered to be defects, with definitions that relate to the purposes of this document and/or obtained from reference sources noted include:

5.2.1.1 *contamination* — three dimensional alien material adhering to a surface. (SEMI Compilation of Terms)

5.2.1.2 *grain boundary* — an interface separating two grains, where the orientation of the lattice changes from that of one grain to that of the other. (ASTM E 7)

5.2.1.3 *inclusion* — indigenous or foreign material within the metal, usually referring to non-metallic

compound particles such as oxides, alumina, sulfides or silicates.

5.2.1.4 *pit* — a surface cavity or crater with a defined edge not caused by impact.

5.2.1.5 *process marks* — a surface texture or pattern that is characteristic of the surface finishing process employed.

5.2.1.6 *scratch* — an elongated mark or groove cut in the surface by mechanical means, not associated with the predominant surface texture pattern. (adapted from SEMI Compilation of Terms)

5.2.1.7 *stringer* — a microstructural configuration of alloy constituents or foreign nonmetallic material, or trace thereof, lined up in the direction of working. (adapted from ASTM E 7)

5.2.2 *wetted surface* — surfaces of a component contacting the contained fluids. (adapted from SEMI Compilation of Terms)

5.2.3 *working distance* — the distance between the surface of the specimen being examined and the front surface of the objective lens. (ASTM E 7)

6 Summary of Method

6.1 Obtain a SEM photomicrograph of a representative area of the wetted surface of the sample at a magnification of 200×. Increase the magnification to 1000× and take another SEM photomicrograph within this area, and another at a pre-selected magnification within the range 3000 to 3600×. Obtain 3000–3600× photomicrographs at two additional representative areas, not necessarily within the 200× magnification region.

6.2 If inclusions or contaminants are noted on any 3000–3600× magnification photos, EDS spectra of representative example(s) may be obtained.

6.3 Perform grid overlay defect counting on the three 3000–3600× photos. Report results in tabular form; provide photomicrographs and EDS spectra with identification of elements detected.

7 Interferences

7.1 Interferences may be encountered as overlaps in EDS spectral peaks. Specific overlaps that may be encountered include but are not limited to:

7.1.1 *Molybdenum and Sulfur* — Overlap in EDS spectra of Mo L peaks and S K peaks prevents unambiguous determination.

7.1.2 *Manganese and Chromium/Iron* — Overlap in EDS spectra of Mn K α peak with Cr K β peak, and Mn

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2 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

3 Institute of Electrical and Electronics Engineers, IEEE Operations Center, 445 Hoes Lane, P.O. Box 1331, Piscataway, New Jersey 08855-1331, USA. Telephone: 732.981.0060; Fax: 732.981.1721