

SEMI F58-1000

TEST METHOD FOR DETERMINATION OF MOISTURE DRY-DOWN CHARACTERISTICS OF SURFACE-MOUNTED AND CONVENTIONAL GAS DISTRIBUTION SYSTEMS BY ATMOSPHERIC PRESSURE IONIZATION MASS SPECTROMETRY (APIMS)

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 28, 2000. Initially available on SEMI OnLine August 2000; to be published October 2000.

1 Purpose

1.1 This document describes the procedure for determination of the moisture dry-down characteristics (quantity of removable moisture) of surface mounted and conventional gas distribution systems (integrated gas distribution systems). APIMS is currently the method of choice for such dynamic tests because it is the commercially available technique capable of ppt moisture analysis with the fastest response time. This test method may provide guidelines for the application of other techniques with similar detection limits and response time to APIMS which are not commercially available at this time.

1.2 The results of this test can be used for qualitative ranking of gas delivery based on the design. It can also be used by a sufficiently sophisticated user as input for numerical simulation of distribution system behavior.

2 Scope

2.1 This test method applies to all types of surface mounted and conventional gas distribution systems used in semiconductor processing.

2.2 *Test Medium* — The test procedure will be carried out in nitrogen. Other “inert” gases will have different purging characteristics and may dry a system more quickly or slowly. Reactive gases may react chemically with moisture. Considerations relating to corrosion resistance are outside the scope of the present document, although the test procedure may prove useful in corrosion studies. The results will provide a ranking with respect to moisture contribution arising as a result of differences in design, which may be applied with due caution to systems intended for use in other gas applications.

2.3 *Operating Situations* — Moisture contribution from a gas delivery system may be the result of contamination arising in its manufacture, or from subsequent exposure to ambient air or non-dry gas. Thus, it is necessary to consider two main situations:

2.3.1 The “initial dry-down” situation, which is determined by the moisture content of the components in the system (as received) with the effects of manufacturing process and design, surface quality, pre-treatment and packaging convoluted together.

2.3.2 The “response to upset” situation, which is determined by the amount of moisture taken up by the system and subsequently released in any exposure after receipt.

2.4 *Safety Issues* — 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 This test method allows the determination of moisture interactions which can be used, for example, to rank systems in order of decreasing moisture interaction. Because different degrees of moisture interaction are permissible in different situations, selecting the “best” system requires consideration of how they will be used, either qualitatively or through a numerical simulation of distribution system behavior. The results of this test can only be used for qualification for gas delivery systems based on different designs. It cannot be used for production purposes and/or certification/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 C15 — Test Method for ppm and ppb Humidity Standards

SEMI F27 — Test Method for Moisture Interaction and Content of Gas Distribution Systems and Components by Atmospheric Pressure Ionization Mass Spectrometry (APIMS)

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

4.2 ASTM Standards¹

ASTM-F1397-93 — Standard Test Method for Determination of Moisture Contribution by Gas Distribution System Components

5 Summary of Test Method

5.1 This test method consists of initial drydown and moisture input tests for both surface-mounted and conventional gas delivery systems.

6 Terminology

6.1 Abbreviations and Acronyms

6.1.1 *APIMS* — Atmospheric Pressure Ionization Mass Spectrometry.

6.1.2 *EPSS* — electropolished stainless steel.

6.1.3 *ppm* — molar parts per million ($\mu\text{mole/mole}$). The same as ppmv.

6.1.4 *ppb* — molar parts per billion (nmole/mole). The same as ppbv.

6.1.5 *ppt* — molar parts per trillion (pmole/mole). The same as pptv.

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

6.2 Definitions

6.2.1 *baseline* — an instrument response under steady state conditions.

6.2.2 *glove box* — an enclosure that contains a controlled atmosphere, usually inert.

6.2.3 *induction time* — the elapsed time between when humidified gas is introduced to the test system and when moisture is detected at the moisture analyzer. For a test system which is perfectly transparent to moisture, the induction time is equal to the residence time of the gas in the system.

6.2.4 *peak height* — the maximum moisture concentration recorded when a moisture input of pre-defined length and concentration is introduced to a test system.

6.2.5 *response time* — the time required for the test system to reach steady state after a change in concentration.

6.2.6 *test system* — the gas delivery system under test

7 Required Equipment

7.1 *APIMS* — The APIMS used for moisture detection can be of any type. Calibration of the APIMS shall be performed as per SEMI F33, one at the beginning and one at the end of the series. These calibrations shall both be made under the same analytical conditions (flow, pressures, plate voltages, etc.) as the tests and the results shall be within 5% of each other. Provided this condition is met, the interval between calibrations may be left to the discretion of the operator; however, it is suggested that calibration should be carried out whenever the equipment is moved and/or every two weeks of operation.

7.2 *Dry Gas and Moisture Generator* — A source of extremely dry nitrogen (less than 200 ppt moisture) and a moisture generator capable of delivering up to 13 slpm nitrogen doped with 200 ppb moisture is required. This generator may be the same as used to calibrate the APIMS. The output of the generator shall be verified according to SEMI C15.

7.2.1 Most ultra-high purity gas panels are currently fitted with metal gasket type connectors. The same type of connector should, therefore, be incorporated into the test bench for connection to the test system.

7.3 *Test Blank* — Any series of tests shall include the results of testing a blank. The blank shall be the shortest convenient length (no more than 1 m) of 1/4" EPSS tubing with suitable fittings and/or adapters at either end to enable it to be inserted in place of the test system.

7.4 *Moisture Pulse Generator* — A valve arrangement capable of switching instantaneously between dry and humidified nitrogen is also required. An example of such a design is shown in Figure 1. In this design, flow is maintained in both the humidified and dry gas lines at all times. By simultaneously switching valves V_1 and V_3 , or V_2 and V_3 , either humidified or dry gas is directed through the test system while the other gas stream is directed to vent. Note that there is a bypass loop so that flow can be maintained to the APIMS when the test system is removed. In this design, all gas lines, but especially those lines between V_1 and V_3 and between V_2 and V_3 , should be as short as possible and constructed of EPSS tubing of high quality. Maintaining gas lines at a constant temperature between 50 and 80°C wherever possible is also recommended.

7.4.1 The bypass loop will contain a stagnant volume of gas during testing. To avoid exposing the APIMS to a large moisture upset whenever it is fed with the gas contained in the bypass loop, the loop should be thoroughly baked out (at $\geq 200^\circ\text{C}$) and protected from atmospheric contamination thereafter. Valve V_4 should

¹ Available from American Society for Testing and Materials, 100 Barr Harbor Dr, West Conshohocken, PA 19428. Fax: 1-610-832-9555. World Wide Web: <http://www.astm.org>.