

SEMI F55-0600

TEST METHOD FOR DETERMINING THE CORROSION RESISTANCE OF MASS FLOW CONTROLLERS

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 April 10, 2000. Initially available on www.semi.org May 2000; to be published June 2000.

1 Purpose

1.1 A mass flow controller (MFC) is often used to control corrosive gases under unfavorable conditions. This test method is intended to help differentiate between MFC designs on the basis of relative resistance to corrosion-induced failure.

2 Scope

2.1 This test is intended to show the effect of corrosion caused when a corrosive gas such as HCl is contaminated by an oxidizer such as atmospheric moisture. For the purpose of this test HCl is the preferred test gas, however this test can also be performed with other gasses. This test method describes a corrosive gas exposure test for mass flow controllers. The test is intended to accelerate the corrosion while simulating conditions that may be found within process equipment and gas systems in the semiconductor industry. As the relationship between corrosion and performance may differ with MFC design, corrosion is not measured directly. The effects of corrosion are detected by observing changes in MFC calibration and other operating parameters.

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 This test method should not be expected to yield comparable quantitative results from one test facility to another.

3.2 Results may be compromised by the methods used to construct the apparatus.

3.3 This test is designed to be a destructive test. The MFC tested will be destroyed. This test is intended only for MFCs manufactured for use in HCl or a similar highly corrosive gaseous environment. MFCs manufactured for non-corrosive service may develop leaks or other catastrophic failures if tested by this method. For practical reasons in constructing and

operating the test bed, test samples may be limited to 100 sccm N₂ equivalent full scale (FS) flow.

3.4 This method does not measure corrosion directly by analyzing or inspecting the gas-wetted surfaces following exposure to a corrosive. No attempt is made to detect particles in the exit gas stream resulting from the corrosion process.

4 Referenced Documents

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

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

5 Terminology

5.1 Acronyms and Abbreviations

5.1.1 *DUT* — device under test

5.1.2 *FS* — full scale

5.1.3 *kPa* — kiloPascal

5.1.4 *MFC* — mass flow controller

5.1.5 *MFM* — mass flow meter

5.1.6 *ppbv* — parts per billion by volume

5.1.7 *ppmv* — parts per million by volume

5.1.8 *sccm* — standard cubic centimeters per minute using a standard temperature of 0° C and a standard pressure of 101.32 kPa.

5.2 Definitions

5.2.1 *actual flow* — for the purpose of this standard, the output value of the reference flowmeter.

5.2.2 *ambient temperature* — the temperature of the medium surrounding the device. Under ordinary laboratory benchtop conditions, ambient temperature is the temperature of the room.

5.2.3 *indicated flow* — the electrical output of the device under test (DUT).

5.2.4 *valve drive* — electrical output from the DUT which is analogous to the level of power supplied to the

control valve. This output is sometimes called valve voltage.

6 Summary of Method

6.1 Corrosive gas and moisture-containing N_2 are alternately flowed through the DUT with intervening dry N_2 purges. The test MFC is monitored over time for changes in performance resulting from corrosion.

6.2 This test is intended to be an accelerated simulation of the effects of alternating corrosive and moisture-exposure conditions. The test records how long it takes for corrosion to affect calibration. MFCs that are more resistant to corrosion failures should last longer under the test conditions. A dry N_2 purge separates each MFC's exposure to the corrosive gas and to the moisture-containing gas. This purging favors MFC designs that perform well in corrosive service because those MFCs can be dried out quickly with limited purging.

6.3 This method is expected to yield data that can be used to compare the relative performance of components tested for the purpose of qualification.

7 Interference's

7.1 Portions of the gas system exposed to HCl may require replacement or cleaning and refurbishment between test sequences to avoid invalidating subsequent tests by prematurely contaminating DUTs. Take care in the design and maintenance of the system so that particles shed from upstream piping components as a byproduct of corrosion are minimized so that they do not affect the DUT.

8 Apparatus

8.1 *Containment System* — to contain corrosive gas leaks that may develop in the DUT or gas system. The portion of the system that contains the corrosive gas should be operated in a suitable secondary containment environment.

8.2 *Power Supply, Controls, and Data Collection Devices* — capable of recording data 20 times per second. Signals should be measured with a resolution of one mV or better. Data may be recorded in convenient units such as volts, sccm, or percent of FS. For the purpose of analysis and reporting, data should be converted to percent of FS except for valve drive, which may be reported in either percent of maximum drive or volts.

8.3 *DUT*

8.4 *Data Acquisition System* — Recommended as a data collection system to facilitate test sequencing and data collection.

8.5 *Plumbing* — with tubing and gas-switching valves constructed from 316L electropolished stainless steel or other material that exhibits superior corrosion resistance compared to 316L. The use of materials such as plastics, which retain or are permeable to water vapor, are not appropriate. All-metal sealed valves (no elastomers) are readily available and must be used.

8.6 The test plumbing is intended to represent the technology level found in the semiconductor process gas piping in which a typical DUT will operate.

8.7 A filter has not been specified immediately upstream of the DUT because it would harbor contaminants and make it difficult to alternate gas types.

8.8 *Dry N_2 Source* — pressure regulated source with a moisture level no greater than 50 ppbv.

8.9 *Dry HCl Source* — pressure regulated source with a moisture level no greater than 500 ppbv.

8.10 *Reference Flowmeter* — traceable with calibration certificate, calibrated for N_2 , FS flow 100% to 125% of the FS N_2 equivalent flow range of the DUT. Accuracy better than 5%, linear to 0.5% FS repeatable to 0.15% over a three-hour period, and reproducible to 0.3% of the test flow for the duration of the test.

NOTE 2: As this test is concerned with changes in DUT calibration and not the value of DUT calibration itself, the accuracy of the reference flowmeter is not paramount.

8.11 *Moisture Generator* — to introduce water vapor in a concentration of 100 ppmv ($\mu\text{L/l}$) $\pm 20\%$ in N_2 .

8.12 *Moisture Analyzer* — to periodically verify the output of moisture generator.

9 Reagents and Materials

9.1 *HCl*

9.2 *N_2*

10 Safety Precautions

10.1 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

NOTE 3: The DUT will be contaminated with corrosive material at the conclusion of this test. Caution should be exercised in handling. A contaminated DUT should be properly decontaminated.

10.2 Become familiar with the safe handling practices associated with the corrosive gas before undertaking this test.

11 Preparation of Apparatus

11.1 The test apparatus is the source of three gases—dry N₂, dry HCl, and N₂ containing a known level of water vapor. Each gas can be valved through the DUT individually with minimal cross contamination. The DUT actively controls the gas flow in each case. The gas then flows into either a scrubbed exhaust system for safe disposal or a reclamation system. A reference flowmeter in the dry N₂ supply line is used to detect changes in calibration of the DUT when dry N₂ is flowing in series through both devices.

11.2 *Setup and Schematic* — See Figure 1 for a schematic of the test apparatus. This schematic may not show in detail all the components required in an actual system.

11.3 Supply the manufacturer's recommended power to the DUT. Provide MFC setpoint and purge command (if available). Monitor the following:

11.3.1 DUT setpoint input.

11.3.2 DUT indicated flow output.

11.3.3 DUT valve drive output (if available)

11.3.4 actual flow (output of reference flowmeter)

11.4 Verify that the proper moisture levels exist in each of the test gases.

11.5 Provide a purge flow loop that maintains a constant flow of dry N₂ in the line from the DUT to the exhaust system. Flow in this loop must be sufficient to prevent back diffusion into the DUT of moisture generated in the exhaust system, particularly when flow into the DUT is shut off for measuring zero.

11.6 If the moisture generator is supplied from the same N₂ source that supplies the dry N₂, take precautions to avoid the diffusion of moisture from the moisture generator into the dry N₂ line. Such precautions may include the use of separate pressure regulators and other valving.

11.7 Moisture generators typically take time to stabilize. A continuous flow should be maintained through the moisture generator during the test to maintain a stable moisture flow. This may require diverting the output flow to a vent when not flowing through the DUT.

11.8 Periodically verify the moisture concentration of wet N₂ with a suitable moisture analyzer.

11.9 Follow manufacturer set-up procedures for all equipment.

11.10 Conduct the test at an ambient temperature of $22 \pm 2^\circ\text{C}$.

11.11 The DUT moisture generator, flowmeters, and other instrumentation should have been stabilized at room temperature for 24 hours before beginning the tests. It is not necessary to flow gas during this stabilization time.

11.12 All portions of the plumbing system that could contain HCl must be helium leak tested per SEMI F1 before introducing HCl into the system. When changing the DUT or other components, at least those portions of the system that were changed must be re-tested.

NOTE 4: Small leaks in a corrosive gas system can become much larger without warning.

12 Calibration and Standardization

12.1 Calibrate all instrumentation using current standards that are traceable to an appropriate standards laboratory. In the absence of appropriate standards, use manufacturer recommendations for calibration.

13 Procedure

NOTE 5: Refer to Figure 2 for a flowchart of the following test.

13.1 Set the pressure of each of the HCl and Dry N₂ gas sources at $140 \text{ kPa} \pm 5\%$. Set the pressure of the N₂ to the moisture generator to the appropriate value recommended by the manufacturer. If the test must be run at some other pressure, because of a limitation of the DUT, report this fact in the test results.

13.2 Start the wet N₂ flow to vent or through the moisture analyzer. Establish a stable moisture concentration at $100 \text{ ppmv} \pm 20\%$ before beginning the test sequence. Set the wet N₂ to flow within $\pm 5\%$ of the DUT flow that will be run during the test.

13.3 Place the DUT control valve in the purge or fully open position by supplying a purge command or a 200% setpoint. Purge with dry N₂ at a rate of one to two times FS flow for one hour to establish initial conditions. The purge rate is controlled by adjusting the supply pressure and is monitored by a purge-rate flowmeter located in the gas-supply path. Follow the manufacturer's procedure for fully opening the DUT control valve.

13.4 Provide a setpoint to the DUT for $80 \pm 5\%$ of FS flow.

13.5 Determining the Test Baseline and Measurement Repeatability.