SEMASPEC Provisional Test Method for Determining the Corrosion Resistance of Mass Flow Controllers

SEMATECH
Technology Transfer 92071233B-STD

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Abstract: This test method is intended to help differentiate between mass flow controller (MFC) designs on the basis of relative resistance to corrosion-induced failure. The method describes a corrosive gas exposure test for MFCs. The test is intentionally aggravated to accelerate the test while simulating conditions that may be found within process equipment gas systems in the semiconductor industry. The MFC is operated during the test. As the relationship between corrosion and performance amy 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. This test is intended only for MFCs manufactured for use in HCl or a similar corrosive gaseous environment. This revision of the document incorporates changes made as a result of industry review and from corrections made during working session four of the MFC Test Methods Development Task Force. This test method is provisional until it has been validated. This document is in development as an industry standard by Semiconductor Equipment and Materials International (SEMI). When available, adherence to the SEMI standard is recommended.

Keywords: Testing, Mass Flow Controllers, Gas Distribution Systems

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1. Introduction

1.1 *Purpose*—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.

1.2 *Scope*—This test method describes a corrosive gas exposure test for mass flow controllers. The test is intentionally aggravated to accelerate the test while simulating conditions that may be found within process equipment 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.

1.3 *Limitations*—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 50 sccm N_2 equivalent full scale (FS) flow.

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

2. Referenced Documents

2.1 Material safety data sheet (MSDS) for hydrogen chloride, which can be obtained from a supplier of the gas.

3. Terminology

3.1 *Acronyms and Abbreviations*

3.1.1 *DUT*—device under test

3.1.2 *FS*—full scale

3.1.3 *kPa*—kiloPascal

3.1.4 *MFC*—mass flow controller

3.1.5 *MFM*—mass flow meter

3.1.6 *MSDS*—material safety data sheet

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3.1.7 *NIST*—National Institute of Standard Technologies

3.1.8 *ppbv*—parts per billion by volume

3.1.9 *ppmv*—parts per million by volume

3.1.10 *sccm*—standard cubic centimeters per minute using a standard temperature of 0 °C and a standard pressure of 101.32 kPa. The common industry usage is equivalent to scm^3.

3.2 *Definitions*

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

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

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

3.2.4 *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.

4. Summary of Test Method

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 resulting from corrosion.

5. Significance and Use

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

6. Apparatus

6.1 *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.

6.2 *Power Supply*, *Controls*, and *Data Collection Devices*, capable of recording data 20 times per minute. 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.

6.3 *DUT*, which may be limited to 500 scm^3 N_2 equivalent FS flow.

6.4 *Automatic Test Sequencer*, recommended with a data collection system to facilitate test sequencing and data collection, due to the length and repetitive nature of the test.

6.5 *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 (see Section 8.2).

The test plumbing is intended to represent the technology level found in the semiconductor process gas piping in which a typical DUT will operate. For that reason, while reasonable care must be taken to avoid generating particles upstream of the DUT, a filter to protect the DUT from particles originating in the plumbing has not been specified.

6.6 *Dry N_2 Source*, with N_2 supplied through a pressure regulator, purge flow meter, and purifier.

6.7 *Dry HCl Source*, with HCl supplied through a pressure regulator and a purifier. One or more valves must be included in the construction of the HCl system (for example, V8 in Figure 1). The valves allow the system to be purged with dry N_2 when gas cylinders or components are replaced.

6.8 *Purifier*, to reduce the level of moisture in N_2 and HCl to less than 10 ppbv (µl/l), supplied with an endpoint detector or other means to verify that its output remains below 10 ppbv.

6.9 *Reference Flowmeter*, NIST traceable, calibrated for N_2, FS flow 100% to 125% of the FS N_2 equivalent flow range of the DUT (for example, when testing a 100 sccm HCl device capable of flowing 100 sccm of N_2 at FS, use a reference flowmeter calibrated for anything from 100 to 125 sccm N_2). 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: 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.]

6.10 *Water Permeation Device*, to introduce water vapor in a concentration of 100 ppmv (µl/l) ± 20% in N_2.

[Note: This device may be described in more detail following test validation.]

[Note: While this test method uses a permeation-type moisture generator, other moisture sources are available. Any of these other sources can be substituted in the test, if its output is monitored with a moisture analyzer often enough to ensure that its output remains within the test specifications.]

6.11 *Moisture Analyzer*, to periodically verify the output of the water-permeation device.

7. Materials

7.1 *HCl*, 99.9% or better.

7.2 *N_2*, 99.99% or better.

8. Precautions

8.1 *Safety Precautions*

8.1.1 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.

[**Warning:** The DUT may be contaminated with corrosive material at the conclusion of this test. Caution should be exercised in handling. A potentially contaminated DUT should be properly decontaminated following failure analysis.]

8.1.2 Review the MSDS for hydrogen chloride before undertaking this test.

8.2 *Technical Precautions*

[**Caution:** This test is inherently destructive to the device under test.]

8.2.1 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.

9. Preparation of Apparatus

The test apparatus is the source of three gases—dry N_2, dry HCl, and N_2 containing a known level of water vapor. Each gas can be valved through the DUT individually with minimal carryover. The DUT actively controls the gas flow in each case. The gas then flows into a scrubbed exhaust system for safe disposal. A reference flowmeter in the dry N_2 supply line is used to detect changes in calibration of the DUT when dry N_2 is flowing in series through both devices.

9.1 *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.

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

DUT setpoint input

DUT indicated flow output

DUT valve drive output (if available)

actual flow (output of reference flowmeter)

Monitor purifier endpoint detectors and other instrumentation.

9.3 Set up automatic gas-switching valves in conjunction with the automatic test sequencer to produce the required test sequence.

9.4 Locate the gas valves close to the DUT. Use at least 10 diameters of straight tubing between the valves and the DUT to reduce upstream piping effects from tubing bends, elbows, etc. Minimize dead legs in the plumbing so that each gas can be valved through the DUT with minimal carry over of the previous gas.

9.5 In the dry HCl supply line, install a means to detect excess HCl flow resulting from the failure of the DUT. Construct the apparatus so that the HCl supply shuts down when an excess HCl flow is detected.

9.6 Provide a purge flow loop that maintains a constant flow of dry N_2 in the line from the DUT to the scrubber. Flow in this loop must be sufficient to prevent back diffusion into the DUT of moisture generated in the scrubber, particularly when flow into the DUT is shut off for measuring zero.

9.7 If the water permeation device is supplied from the same N_2 source that supplies the dry N_2 purifier, take precautions to avoid the diffusion of moisture from the permeation device into the purifier. Such precautions may include the use of separate pressure regulators and other valving.

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9.8 Provide a flow controller in a vent and moisture test line from the permeation humidifier so that the same flow rate under which the DUT test will be conducted can be maintained while venting flow or conducting a moisture test. The flow controller must be located downstream of the permeation device and down stream of the line which is valved to the DUT so that the pressure in the permeation device remains constant regardless of where the gas is flowing. Program the test sequencer to run the wet N_2 to the vent or the moisture test port whenever it is not switched to the DUT. This has the effect of maintaining the permeation humidifier under the same pressure and flow conditions for the duration of the test. Periodically verify the concentration of wet N_2 with a suitable moisture analyzer. Periodically verify that the permeation device contains the proper amount of water.

9.9 Follow manufacturer set-up procedures for all equipment.

10. Calibration and Reference Standards

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

11. Conditioning

11.1 Conduct the test at an ambient temperature of 22 ± 2 °C.

11.2 Gas temperature must be at ambient temperature ± 1 °C. All test conditions shall remain within manufacturer's specifications.

11.3 The DUT humidifier, 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.

12. Procedure

[Note: Refer to Figure 2 for a flowchart of the following test.]

12.1 *Installing the DUT and Preparing to Start Test Run*

12.1.1 Install the DUT and apply power.

12.1.2 All portions of the plumbing system that could contain HCl must be helium leak tested to a minimum inboard leak level of 1×10^{-7} atm-cc/sec (He) 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 retested.

[**Warning:** Small leaks in a corrosive gas system can become much larger without warning.]

12.1.3 Set the pressure of each of the three gas sources at 140 kPa ± 5%. If the test must be run at some other pressure, because of a limitation of the DUT, report this fact in the test results.

12.1.4 Start the wet-N_2 flow to vent or through the moisture analyzer. Establish a stable moisture concentration at 100 ppmv ± 20% before beginning the test sequence. Set the wet N_2 to flow within ± 5% of the DUT flow that will be run during the test.

12.1.5 Place the DUT control valve in the purge or fully open position by supplying a purge command or a 200% setpoint. Purge with dry N_2 at a rate between FS flow and 1000 sccm 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.

12.1.6 Provide a setpoint to the DUT for 80 ± 5% of FS flow. With dry N_2, stabilize flow and DUT temperature for one hour or for the manufacturer's warm up time, whichever is longer.

12.2 Determining the Test Baseline and Measurement Repeatability

[Note: Data throughout this test may be recorded in a format similar to that found in Table X1.1.]

12.2.1 Open valve V1 and flow dry N_2 at a setpoint of 80% ± 5% of the DUT FS and wait 10 minutes.

12.2.2 Record:

1. DUT setpoint
2. DUT indicated flow
3. DUT valve drive output (if available)
4. Actual flow

12.2.3 Stop flow by closing V1. Drive the MFC control valve to a closed position by providing a zero setpoint or as otherwise provided by the manufacturer. Allow the MFC indicated flow output to stabilize for a minimum of three minutes.

12.2.4 Record the DUT indicated flow and the actual flow during the zero flow condition.

12.2.5 Repeat steps 12.2.1 through 12.2.4 twelve times. This will yield 12 data points for each of the six measured parameters.

12.2.6 Close V1 to stop the dry N_2 flow.

12.2.7 For each of the six parameters recorded, perform the following calculation on the 12 baseline data points to determine the repeatability of the measurement:

$$\frac{(\text{highest value} - \text{lowest value}) \times 100}{\text{average of the 12 values}}$$

If each of the six parameters is not repeatable within 0.2% during these two hours, subsequent test data may not be valid and the problem should be resolved before proceeding with the test.

12.2.8 Baseline

[Note: Use the averaged value of the measurements made in Section 12.2 for each parameter as the baseline for that parameter in the following tests.]

12.3 *Test Sequence*—Without changing the DUT setpoint, repeat the test sequence in Sections 12.3.1 - 12.3.4 every 30 minutes, continuously, 24 hours per day, seven days per week until one of the following test termination conditions is met.

The test should not be interrupted because the DUT may continue to corrode during the interruption, making test results nonrepeatable. The output of the water permeation device should be measured periodically during the test sequence to ensure that it remains within the test specifications.

Terminate the test sequence if:

The DUT fails to function

The actual-flow shift from baseline exceeds 25%

The valve drive exceeds the manufacturer's limits

The DUT indicated flow differs from commanded setpoint by > 5%

The test exceeds 5000 cycles

12.3.1 Flow dry N_2 for five minutes. After five minutes, record data on the six test parameters as done in Section 12.2.7, interrupting flow to record the DUT indicated flow and reference flowmeter output at zero flow. After taking measurements, flow dry N_2 again so that the total length of this step is 10 minutes. Record data as shown in Figure 3.

12.3.2 Flow dry HCl for seven minutes.

12.3.3 Repeat Section 12.3.1 and then proceed to Section 12.3.4.

12.3.4 Flow wet N_2 for three minutes. If no test termination condition is met as described in Section 12.3, repeat Sections 12.3.1 - 12.3.4. If a test termination condition is met, proceed to Section 12.4.

12.4 After the test has been completed, place the DUT control valve in the purge or fully open position. Post-test purge with dry N_2 for one hour or at least 10 volume changes. Cycle the purge gas for 15 seconds on and 15 seconds off by opening and closing V1 for the duration of the purge. Follow the manufacturer's procedure for fully opening the DUT control valve.

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[**Caution:** Verify purge flow with the reference flowmeter or by using other means. Corrosion may have rendered the control valve inoperable, and alternate approaches for purging any corrosive material from the system may have to be followed.]

12.5 *Alternate Approach to Post-Test Purging*

12.5.1 If gas cannot be purged through the DUT, cycle-purge each end of the DUT separately using Sections 12.5.2 through 12.5.4.

12.5.2 With valves V2 and V3 closed for the duration of the procedure, close valve V6 and pressurize both sides of the system by opening V1 and V7 for 15 seconds.

12.5.3 Vent pressure through the scrubber by closing V1 and opening V6 for 15 seconds.

12.5.4 Repeat pressure and vent cycles for a minimum of one hour.

12.6 Remove the DUT. Preserve the test system, if desired, by taking steps to exclude atmospheric moisture. Such steps could include capping the system and shutting all valves. Alternately, the DUT may be replaced with a spool piece. The system can then be continuously purged with dry N_2.

[**Warning:** The DUT may be contaminated with corrosive material. See *Precautions* in Section 8. Preserve the DUT for failure analysis, if desired.]

13. Data Presentation

13.1 Report data in a tabular format and graph per Appendix X1. Any parameter which changes more than 1% from its baseline should also be graphed. Note the reason the test was terminated. Test results may be summarized in a single parameter by reporting the number of test cycles to reach a 2% shift in actual flow or to reach DUT failure, whichever occurs first. In this test, more data is reported than is required to calculate the gross test result. The extra data is used when analyzing test results to determine the cause of failure. Further explanation of the significance of the data is beyond the scope of this test method.

13.2 *Data analysis*. Calculate any parameter-shift in percent as follows:

$$\frac{(\text{baseline value - current value}) \times 100}{\text{baseline value}}$$

14. Precision and Bias

This method is limited in its precision by the calibration of the measurement devices, the temperature of the apparatus, and the concentration of water vapor in N_2. Sources of bias are unknown at this time, but they probably include peculiarities in the construction of the apparatus.

15. Illustrations

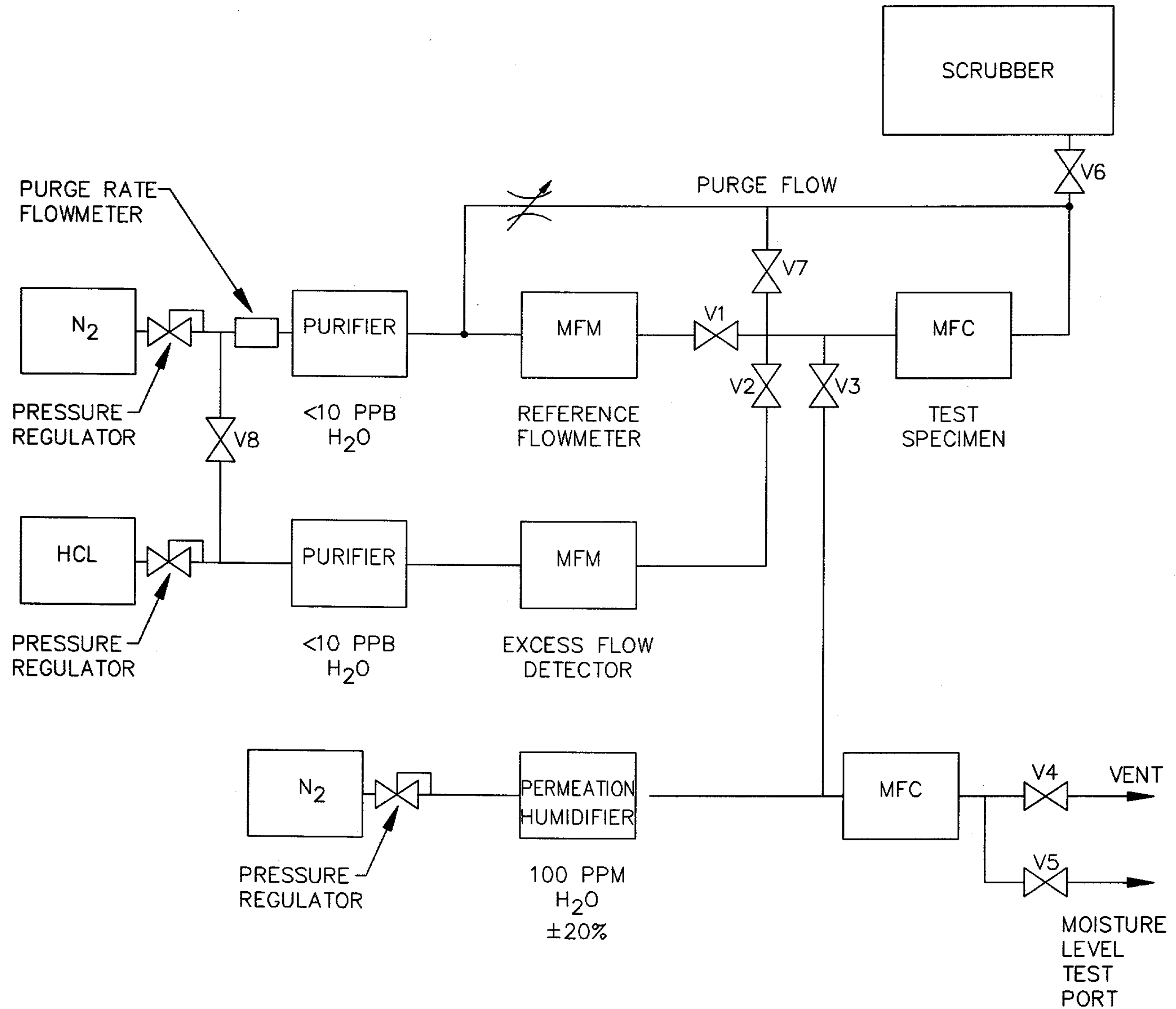

Figure 1 Schematic of Apparatus

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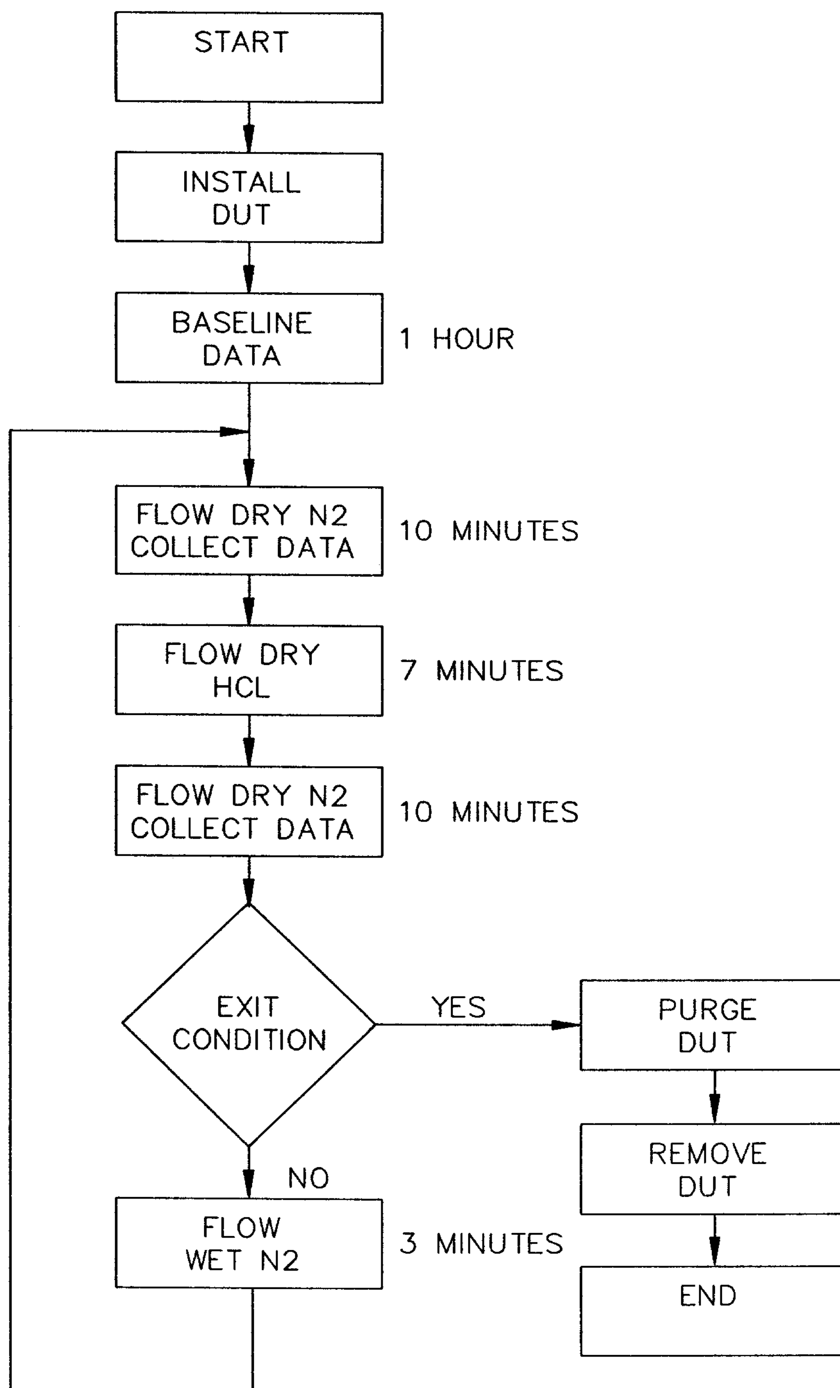

Figure 2 Test Flowchart

(Supplementary Information)

X1 Test Report for SEMASPEC Test Method 92071233B-STD Corrosion Test

MFC Identification: Wxyz Instruments, Model 123, Serial 230967

Calibrated for: 100 scm^3 HCl

Reference Flowmeter: Mnop Company, Model 1023, Serial 37863, 100 sccm N_2

Test dates: March 28 to May 5, 1993

Test Laboratory: Flow Test and S.D. Associates, Inc.

Test supervised by: George Smith

Table X1.1 Data in tubular format

A	B	C	D	E	F	G	H	J
TEST CYCLE #	TEST CYCLE HOURS	DUT SET POINT%	DUT INDICATED %	DUT ZERO %	REFERENCE FLOW ZERO %	ACTUAL FLOW %	CALIBRATIO N SHIFT % CHANGE	VALVE DRIVE VOLTS
base line	base line	75.04	75.05	-0.13	0.03	71.12	0.0	-7.062
100	50	75.01	74.8	0.14	0.04	71.56	0.6	-7.066
200	100	75.00	74.82	0.13	-0.04	71.38	0.4	-7
300	150	74.99	75.1	-0.01	-0.03	71.26	0.2	-7.056
400	200	75.02	75.01	0.02	0.01	71.3	0.3	-7.049
500	250	74.98	74.96	-0.01	-0.03	71.36	0.3	-7.009
600	300	75.02	74.96	0.00	0.03	71.18	0.1	-6.997
700	350	74.99	74.92	0.11	-0.04	71.32	0.3	-7.042
800	400	75.00	75.15	-0.06	-0.04	71.37	0.4	-7.010
900	450	75.02	74.9	0.04	0.01	71.69	0.8	-6.997
1000	500	74.99	75.01	-0.09	0.04	71.96	1.2	-7.056
1100	550	75.01	74.82	0.13	0.04	72.55	2.0	-7.136
1200	600	75.03	75.1	-0.13	-0.02	72.92	2.5	-7.100
1300	650	75.05	75.19	-0.15	-0.03	74.17	4.3	-7.277
1400	700	74.96	75.09	-0.12	-0.01	76.15	7.1	-7.348
1500	750	75.04	75.05	-0.09	-0.01	79.86	12.3	-7.666
1600	800	74.96	75.15	-0.13	0.05	88.36	24.2	-8.278
1700	850	74.96	74.99	0.03	-0.02	110.53	55.4	-9.732

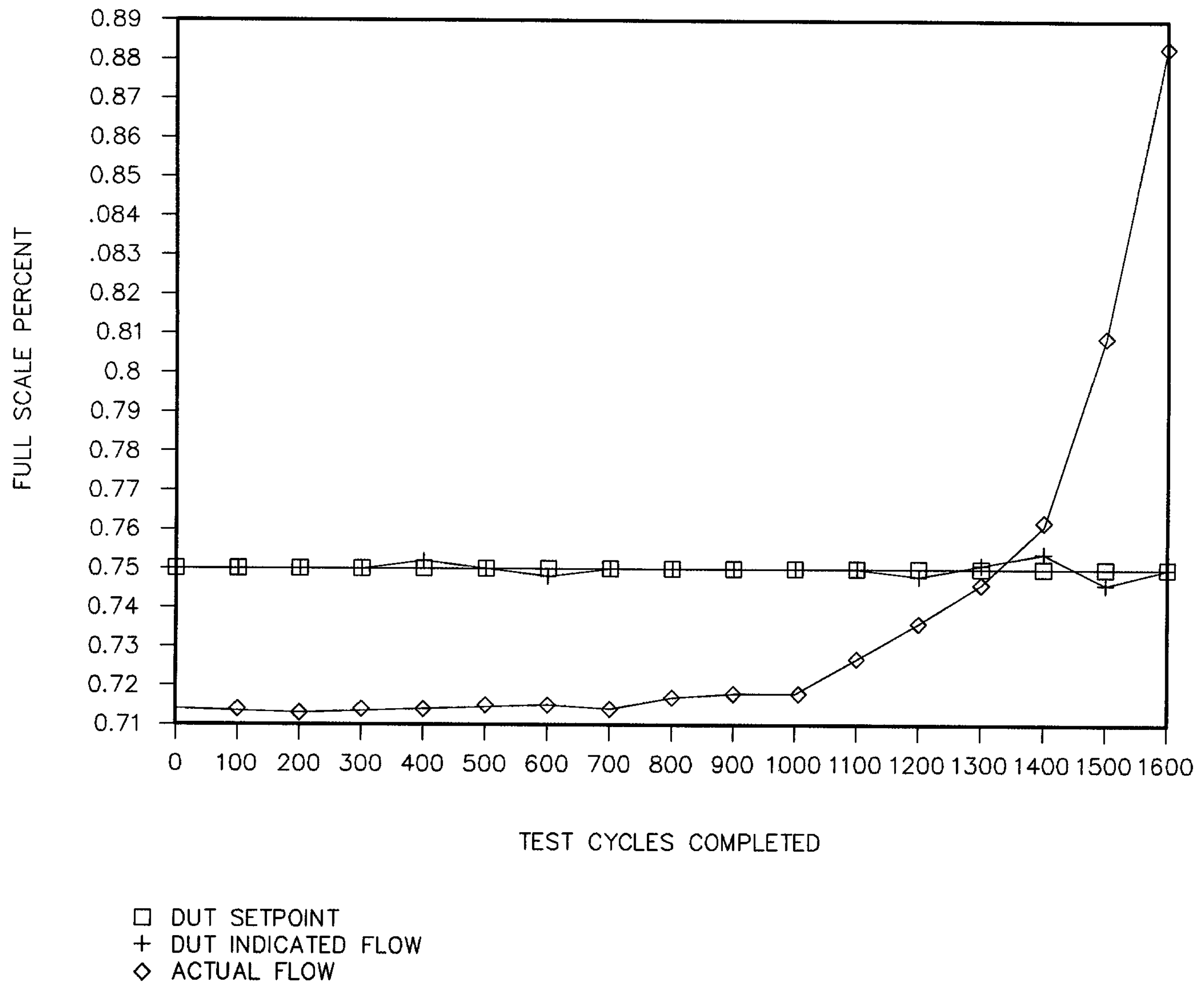

Figure X1.1 Test Results Graph

Appendixes

(Supplementary Information)

X2 Rationale

Corrosion of internal MFC surfaces by the process gas stream is a major cause of MFC failure and the resulting equipment downtime in the semiconductor industry. Corrosion may induce shifts in calibration and in control valve characteristics of the MFC at first, and may eventually lead to complete device failure. Corrosive gases attack the MFC only when they can chemically dissociate in the presence of moisture. Common sources of moisture within semiconductor gas systems are the surrounding atmosphere, the corrosive gases themselves, and impure purge gases. Moisture from the atmosphere can enter through leaks in the gas system (including permeation through elastomers) or when the system is opened to the surrounding atmosphere for maintenance. Even though moisture containing gases and corrosive gases may not intentionally be mixed together, traces of either may remain in the MFC, even after purging. Subsequent exposure of the MFC to the other gas allows the corrosion reaction to proceed. Reducing moisture in corrosive gas systems to the point where corrosion is not a problem requires significant expenditures and continuing operational discipline.

Corrosion becomes significant only when the performance of the MFC is affected. It may commonly occur in some parts rather than in others. One such part is the sensor, which normally operates at an elevated temperature. Another part is the control valve, which may have small orifices. However, equipment design can minimize the effects of corrosion on performance. For this reason, results from conventional tests, such as those subjecting component parts to moist corrosive vapors and looking for visible evidence of attack, may not be sufficient to evaluate a design. More thorough tests are required.

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