

SEMI E108-0307

TEST METHOD FOR THE ASSESSMENT OF OUTGASSING ORGANIC CONTAMINATION FROM MINIENVIRONMENTS USING GAS CHROMATOGRAPHY/MASS SPECTROSCOPY

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1 Purpose

1.1 The purpose of this Standard is to define a test method for the determination of the outgassing organic contamination from minienvironments used for storage and transport of wafers using gas chromatography/mass spectroscopy (GC/MS).

1.2 This Test Method is intended as an alternative to SEMI E46. The main difference between SEMI E46 and this Standard is that SEMI E46 defines a test method which is based on ion mobility spectroscopy (IMS) as the measurement technique while this Standard is based on GC/MS in combination with thermal desorption. Additionally, this Test Method provides a procedure for testing the outgassing of organic compounds in a complete minienvironment. The results of SEMI E46 and this Standard are given in different units.

2 Scope

2.1 The test method provided in this Standard is applicable to the assessment of the outgassing of organic contamination from minienvironments.

2.2 GC/MS is chosen as the method to determine organic contamination because it is commonly used for characterization and quantification of organic compounds. In combination with thermal desorption, it provides a method for the identification of organic compounds in the atmosphere (i.e., inside the minienvironment) as well as directly from source materials, and transferred contaminants. This method can also be used to evaluate materials and processes used in semiconductor industry.

2.3 This Test Method is based on ASTM F1982.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 The test methodologies, metrics and applications provided in this Standard are limited by the following constraints:

3.1.1 The specific recovery of compounds by the proposed standard method strongly depends on the setup of the apparatus used. This has been taken into account by the use of the 'reference-cocktail' (refer to § 9.2).

3.2 Identification of the source of organic compounds inside the minienvironment is out of the scope of this Test Method. For a procedure for the analysis of outgassing organic compounds from individual materials using GC/MS (refer to IDEMA M11-99).

3.3 This Test Method does not provide a procedure how to use the obtained data for assessing the risks that come from individual compounds.

4 Referenced Standards and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI E46 — Test Method for the Determination of Organic Contamination from Minienvironments

SEMI F21 — Classification of Airborne Molecular Contaminant Levels in Clean Environments

4.2 ASTM® Standard¹

ASTM F1982 — Standard Test Methods for Analyzing Organic Contaminants on Silicon Wafer Surfaces by Thermal Desorption Gas Chromatography

4.3 IDEMA Standard²

IDEMA M11-99 — General Outgas Test Procedure by Dynamic Headspace Analysis

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

5 Terminology

5.1 Definitions

5.1.1 *analytical environment* — environment where all analytical measurements are taking place.

5.1.2 *headspace sampling* — in this Standard defined as: collecting volatile organic compounds in an enclosed volume by means of a silicon wafer or silicon wafer chips.

5.1.3 *minienvironment* — a localized environment created by an enclosure to isolate the product from contamination and people.

5.1.4 *sample* — wafer or wafer chips used for the headspace sampling of organic contaminants.

5.1.5 *static storage conditions* — conditions excluding any active movement of test specimens.

5.1.6 *thermal desorption tube* — Analytical equipment capable of collecting organic compounds of interest (i.e., adsorbent filled glass tube).

5.1.7 *wafer* — object made of semiconducting material to be processed, handled or stored in the minienvironment to be tested (i.e., prime or processed silicon wafer).

6 Summary of Method

6.1 This Test Method comprises storage tests of wafers in minienvironments under static storage conditions. The contamination is directly measured from the silicon wafer surface. Three important aspects are covered:

1. Contamination due to the minienvironment alone,
2. Contamination from the use of minienvironments for wafer processing, and
3. Contamination from future materials to be used in semiconductor technology.

6.2 The setup for each test comprises an analytical equipment in the analytical environment as well as a test equipment in the test environment. Each environment has to fulfill the following requirements:

6.2.1 *Analytical Environment* — Shall be clean enough with respect to airborne organic contamination: recommendation is less than 100 pptM for organic compounds with boiling points >150°C (Class MC-100, according to SEMI F21).

¹ ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: +1.610.832.9585, Fax: +1.610.832.9555, <http://www.astm.org>. ASTM trademark is owned by ASTM International.

² The International Disk Drive Equipment and Materials Association, 1226 Lincoln Avenue, Suite 100, San Jose, CA 95125, USA; Telephone: +1.408.294.0082, Fax: +1.408.294.0087, <http://www.idema.org>

6.2.2 *Test Environment* — Shall equal the environment in which the test specimen will be used.

6.3 Silicon wafers are placed in the minienvironment or used for headspace sampling experiments. These wafer samples are then analyzed by GC/MS to determine the amount of contaminants.

6.4 The wafers or wafer chips to be used for the test method are decontaminated as described in ASTM F1982 for bare silicon wafers. The surface condition of the wafer (hydrophobic or hydrophilic) has to be adjusted in a reproducible way and has to be the same for all comparative measurements.

6.5 The wafer or the wafer chips are placed in the minienvironment and are left there under static conditions for the chosen static storage time (depending on the intended use of the minienvironment to be tested, a time between 1 h and 28 d may be used). When loaded into the minienvironment the wafer may have a temperature which reflects production circumstances (i.e., 80°C when unloading from furnace processes). In some situations wafers may be exposed to severe thermal conditions. The minienvironment should be evaluated under the conditions exposed to. Be sure that the material to be tested does not undergo phase transitions under the chosen temperature conditions.

6.6 After the static storage test period, the wafer is returned to the analytical equipment and the organic contamination on the wafer is measured immediately.

6.7 The quantitative value for the total amount of outgassed and adsorbed organics from the minienvironment is calculated as the difference between the detected amount of contamination on a blank sample vs. the amount on the exposed sample.

7 Interferences

7.1 The presence of organic contamination in the atmosphere of the test environment may lead to a significant contribution to the detected total amount of organic compounds on the test wafers or wafer chips. Care has to be taken when subtracting the blank value from the test value. Transport times from test environment to analytical environment have to be as short as possible.

8 Apparatus

8.1 For the analysis of organic compounds on wafers a GC/MS apparatus with thermal desorption unit is required: A gas chromatography (GC) instrument, which utilizes a capillary column to separate a wide variety of organic compounds, combined with mass spectrometer (MS). A thermal desorption unit is used to desorb organics from sample thermal desorption tubes and collect them in a trap. Two types of trapping may be used:

1. cooling the trap down to a minimum of -150°C does not require any adsorbent (glass wool may be used) or
2. a Tenax[®] filled trap has to be cooled down to a minimum of -25°C .

8.1.1 The thermal desorption unit is coupled to the GC instrument via a heated transfer line. This apparatus may be used directly for the desorption from wafer chips which are put in empty glass (or stainless steel) thermo desorption tubes (method A).

8.1.2 A desorption unit for complete wafers is specified in ASTM F1982. In this case sample thermal desorption tubes packed with adsorbent have to be used to trap compounds desorbed from the wafer. These thermal desorption tubes are analyzed within the thermal desorption unit (method B).

8.2 The analysis of outgassing organic contamination from minienvironments requires an extremely sensitive analytical equipment. Recovery and limit of detection for the different classes of compounds must be evaluated carefully in order to ensure reliable results.

9 Reagents and Materials

9.1 For the materials used for handling and transport of wafers clean, decontaminated equipment has to be used (e.g., tweezers heated in a propane flame). Refer to the reagent and materials described in SEMI E46 and ASTM F1982.

9.2 *Test Mixture* — For the calibration of equipment and the quantification of the amount of contamination a test mixture ('cocktail') is used. This mixture was chosen in order to give an 'average' over typical substance classes present in semiconductor production lines, which have high sticking factors to the wafer surface. The response factor of the MS-detection for organics varies significantly. As this is true for all contaminants, the method gives a realistic trustworthy quantitative correlation to a 'typical average compound mixture' of contaminants on wafer surfaces.