

SEMI E46-0307

TEST METHOD FOR THE DETERMINATION OF ORGANIC CONTAMINATION FROM MINIENVIRONMENTS USING ION MOBILITY SPECTROMETRY (IMS)

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NOTICE: This Standard or Safety Guideline has an Inactive Status because the conditions to maintain Current Status have not been met. Inactive Standards or Safety Guidelines are available from SEMI and continue to be valid for use.

1 Purpose

1.1 The purpose of this Test Method is to provide an analytical procedure—ion mobility spectrometry (IMS)—for the determination of organic contamination from minienvironments which has the capability of testing their construction material.

1.2 This Test Method is intended as an alternative to SEMI E108. The main difference between SEMI E108 and this Standard is that SEMI E108 defines a test method which is based on gas chromatography/mass spectrometry in combination with thermal desorption as the measurement technique while this Standard is based on ion mobility spectroscopy. The results of SEMI E108 and this Standard are given in different units.

2 Scope

2.1 Silicon wafers passed through or stored in minienvironments may be affected by organic contamination originating from construction materials. Knowledge of this contamination assists the decision about the application of minienvironments in semiconductor manufacturing.

2.2 IMS was chosen as the method to determine this contamination because it provides an easy, widely applicable, fast and sensitive way to measure organic contamination on surfaces.

2.3 Furthermore, IMS provides the possibility of checking the contaminating effects of processing, chemical carryover, and the characterization of future polymeric materials for use in semiconductor technology.

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3 Referenced Standards and Documents

3.1 SEMI Standards and Safety Guidelines

SEMI E19 — Standard Mechanical Interface (SMIF)

SEMI E108 — Test Method for the Assessment of Outgassing Organic Contamination from Minienvironments using Gas Chromatography/Mass Spectroscopy

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

4 Terminology

4.1 Abbreviations and Acronyms

4.1.1 DOP — dioctylphthalate

4.1.2 HPB — hexaphenylbenzene

4.1.3 IMS — ion mobility spectrometry

4.1.4 IMS/MS — ion mobility spectrometry/mass spectrometry

4.1.5 NS — standardized ground joint

4.1.6 *PFA* — polyfluoroalkoxy

4.1.7 *PP* — polypropylene

4.2 Definitions

4.2.1 *headspace* — the volume above the sample containing the gas to be analyzed.

4.2.2 *standard mechanical interface (SMIF)* — the interface plane between a pod and another minienvironment as per SEMI E19.

5 Summary of Method

5.1 This Test Method defines a fast, sensitive technique for the determination of organic contamination from minienvironments. The contamination is measured directly from the silicon 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.

5.2 Silicon wafers are either placed in the minienvironment or used for headspace sampling experiments. The sample is heated and the target compound is either desorbed or outgassed. These compounds are swept by the carrier gas into the Ion Molecule Reactor. Here the molecules are chemically ionized under atmospheric pressure. The target ions are separated in a drift cell by electrophoreses in the gaseous state and detected by an electrometer. Additionally, a quadrupole mass spectrometer could be used as a second detector. The result is a quantitative value for the total amount of organic surface contamination.

6 Interferences

6.1 To adequately measure organic contaminants from minienvironments, an extremely sensitive method for analyzing volatiles is required. At this time, the only analytical method meeting these requirements is IMS. The absolute determination of organic contamination by the IMS method is still limited, especially in the presence of many analytes, but this proposed test method permits the comparison of quantitative results from different laboratories.

7 Apparatus

7.1 *Minimum Requirements for the Ion Mobility Spectrometer* — For the successful performance of the experiments using the IMS instrument, the following requirements are essential:

- minimum length of the drift cell: 4 cm
- sample inlet has to be lockable (no sniffing devices)
- sample desorption oven must have a programmable temperature control
- the electronics must guarantee a linear amplification of the observed signal
- intensity over at least five orders of magnitude
- documentation of all single measurement results

7.2 *Measuring Parameters* — For measurements with the ion mobility spectrometer, the following parameters have to be used:

- temperature of the desorption furnace: 200°C (specified)
- temperature of the drift cell and IMS detector: 205°C (specified)
- maximum dwell time or channel length corresponding to minimum time resolution: 80 µs (suggested)
- measuring time per spectrum: 40 ms (for 200 V/cm and 10 cm length of drift cell) (specified)
- gatewidth: 200 µs (suggested)

- number of scans: 2000 (suggested)
- drift gas and carrier gas: zero grade (synthetic) air (specified)
 - approx. 10 ppm H₂O (specified)
 - max 0.1 ppmv total hydrocarbons (specified)
- carrier gas flow: 100 mL/min (for a diameter of 3.9 mm) (specified)
- drift gas flow: 500 mL/min (for a diameter of 4.25 cm) (suggested)

8 Reagents

8.1 The chemicals must be of the described quality or better and supplied with a certificate of analysis.

8.1.1 Benzene: ultrapure quality for trace residue analysis. Minimum quality: pro analysi.

- content of benzene: min. 99.8%
- content of water: max. 0.02%
- nonvolatile residue: max. 0.0005%

8.1.2 Ethanol: ultrapure quality for trace residue analysis. Absolute.

- content of ethanol: min. 99.8%
- content of water: max. 0.1%
- nonvolatile residue: max. 0.0005%

8.1.3 DOP: normal pure quality.

- content of DOP: better than 96%

8.1.4 HPB: normal pure quality.

- content of HPB: better than 96%

9 Safety Precautions

9.1 All preparation and measurement work has to be done according to local regulations for laboratories.

10 Procedure

10.1 *Preparation Tools and Auxiliaries* — The following equipment is needed for the preparation of polymer headspace substrates:

- protective eye wear
- 1 fume cupboard
- 1 butane burner
- 2 pairs of crucible tongs made of stainless steel
- scalpel with changeable blades
- 4 pairs of cross tweezers with tips made of steel
- 1 metal vernier caliper
- Si-wafer-chips: formate $20 \times 10 \text{ mm}^2$
- watch glass
- weighing bottle with lid, both made of glass
 - inner diameter: 80 mm