SEMI MF1982-0714
TEST METHODS FOR ANALYZING ORGANIC CONTAMINANTS ON SILICON WAFER SURFACES BY THERMAL DESORPTION GAS CHROMATOGRAPHY

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NOTICE: This Document was completely rewritten in 2014.

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1 Purpose

1.1 Organics are present in many materials, such as plastics, lubricants, cleansers, soaps, and living tissues. Some of these compounds are volatile and others can become airborne through chemical reactions, heating, abrasion, or outgassing. Organic compounds are present in many materials such as parts and components for cleanrooms, carriers, FOUP, developers, and organic removers. Some of these organic compounds are volatile and some can be discharged into clean room environment through chemical reactions or by heating. Also they can transfer to wafers by direct contact or be left behind from solvent residues. Once present in clean facilities, they can deposit on wafer surfaces. The desorbed organic compounds can be transferred to wafers or reticles through the environment. Organics deposited on wafers can cause degradation haze, wafer surface tension changes, irregular oxidation rates, and other effects, such as counter-doping by organophosphorus compounds. Various organic compounds on wafer surfaces can cause a pseudo increase of the thickness of the native oxide film. Nonuniformity of the surface leads to nonuniform oxide on a wafer and causes degradation of the oxide breakdown voltage. It also causes not only wafers, but also the reticles or mirrors to haze. Identification of trace level organic contaminants is important in determining the source of the particular contamination. These test methods use the thermal desorption gas chromatography mass spectrometry (TD/GC-MS)[1],[2] technique to characterize and quantify organics deposited on wafer surfaces. This is one of the reasons that control of organic compounds is required in a cleanroom environment or on wafer surfaces. This mandates test methods of organic compounds on wafer surfaces. TD/GC-MS is useful for qualitative and quantitative analysis of trace-level organic compounds adsorbed on wafer surfaces.

1.2 Monitoring of organic contamination on wafer surfaces also can be used to measure material outgassing for proper selection of cleanroom, construction, and wafer packaging materials.

2 Scope

2.1 This Test Method covers the identification and quantification of organic compounds on wafer surfaces by using gas chromatography mass spectrometry (GC-MS).

2.2 This Test Method describes the procedures for sample preparation and analyses by thermal desorption gas chromatography (TD-GC).

2.3 The range of lower detection limits of this Test Method depends on the species of organic compound. However, generally for organic compounds, it ranges from subpicogram to nanogram per square centimeter of silicon-wafer surface (pg/cm^2 to ng/cm^2). The lower detection limit when the molecular weight is assumed as 300 and carbon numbers as from 10 to 20 is in the range of 5e10–5e11 molecules/cm^2, 1e12-1e13 C atoms/cm^2.

2.4 This Test Method can be used for various materials depending on the purposes, but mainly for bare silicon wafers.

2.5 When testing wafers, full wafers are basically used. However, cleaved wafers can be accepted as long as it does not cause a significant drop in the sensitivity (from a quarter to a half of a wafer).

[1] Fergason, L.A., "Analysis of Organic Impurities on Silicon Wafer Surfaces." *Microcontamination,* April 1986: pp. 33–37.

[2] Saga, K. and Hattori, T., "Identification and Removal of Trace Organic Contamination on Silicon Wafers Stored in Plastic Boxes." *Journal of Electrochemical Society* (1996): pp. 3270–3284.

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3 Limitations[3]

3.1 Some organic compounds show difficulty in measuring by TD/GC-MS. These are volatile compounds, amines, polar compounds (fatty acids, etc.), and high-polymer organic compounds that degraded at 400°C or below. In the TD/GC-MS measurement above, high-polymer organic compounds or polar organic compounds tend to remain in the column or show themselves as broad peaks. Organic materials degraded at 400°C or below may be measured as other materials generated after the degradation. As organic phosphorus tends to remain and degrade, a combination of qualitative analysis by TOF-SIMS and quantitative analysis by inductivity coupled plasma mass spectrometry (ICPMS) after organophosphorus degradation is desirable. For amines, qualitative and quantitative analysis by capillary electrophoresis (CE) or capillary electrophoresis time-of-flight mass spectrometry (CE/TOF-MS) is preferable.

4 Referenced Standards and Documents

4.1 *SEMI Standards and Safety Guidelines*

SEMI M59 — Terminology for Silicon Technology

4.2 *ASTM Standard*[4]

ASTM D6196 — Practice for Selection of Sorbents and Pumped Sampling/Thermal Desorption Analysis Procedures for Volatile Organic Compounds in Air

4.3 *ISO Standards*[5]

ISO/DIS 14644-8 — Classification of Air Cleanliness by Chemical Concentration

ISO/FDIS 14644-10 — Classification of Surface Cleanliness by Chemical Concentration

4.4 *JACA Standard*[6]

JACA No.43-2006 — Analysis of Surface Molecular Contaminants

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

5 Terminology

5.1 General acronyms, terms, and symbols related to silicon technology are listed and defined in SEMI M59.

5.2 *Abbreviations Specific to this Standard*

5.2.1 *C16* — n-hexadecane, n-$C_{16}H_{34}$

5.2.2 *C20* — n-eicosane, n-$C_{20}H_{42}$

5.2.3 *CE* — capillary electrophoresis

5.2.4 *CE/TOF-MS* — capillary electrophoresis time-of-flight mass spectrometry

5.2.5 *GC* — gas chromatography

5.2.6 *GC-MS* — gas chromatography mass spectrometry

[3] T. Taira, S. Hiroshima, Y. Shiramizu et.al "Study of organic contaminants analysis using TD/GC-MS on silicon wafer surfaces" ECS Trans., Vol 16 (6), (2008): pp. 237–48.

[4] 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

[5] International Organization for Standardization, ISO Central Secretariat, 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland; Telephone: +41.22.749.01.11, Fax: +41.22.733.34.30, http://www.iso.org

[6] Japan Air Cleaning Association, Itobeni-Hamacho Building, 2-7-5 Nihonbashi-hamacho, Chuohku, Tokyo, 103-0007 Japan; Telephone: +86.3.3665.5591, Fax: +86.3.3665.5593, http://www.jaca-1963.or.jp

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