

SEMI E45-1101

TEST METHOD FOR THE DETERMINATION OF INORGANIC CONTAMINATION FROM MINIENVIRONMENTS USING VAPOR PHASE DECOMPOSITION-TOTAL REFLECTION X-RAY SPECTROSCOPY (VPD/TXRF), VPD-ATOMIC ABSORPTION SPECTROSCOPY (VPD/AAS), OR VPD/INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (VPD/ICP-MS)

This test method was technically approved by the Global Metrics Committee and is the direct responsibility of the European Equipment Automation Committee. Current edition approved by the North American Regional Standards Committee on August 27, 2001. Initially available at www.semi.org September 2001; to be published November 2001. Originally published in 1995; previously published March 2001.

1 Purpose

1.1 This test method provides the analytical procedures to determine the level of inorganic contamination from a minienvironment.

2 Scope

2.1 This document relates to inorganic impurities, which includes metallic contaminants, whether they occur as atoms, molecules, or particles. The number of metals to be analyzed is restricted to the four elements sodium (Na), calcium (Ca), iron (Fe), and copper (Cu) in order to rapidly characterize minienvironments from a practicable point of view. While Na, Ca, and Fe represent one ensemble of highly detrimental impurities with respect to contamination from human sources (Na), the environment (Ca), or from equipment and corrosive effects (Fe), Cu is analyzed due to its increasing importance in semiconductor manufacturing. Additionally, they are easily analyzed with sufficiently low detection limits. It is up to the user of this test method to quantify additional elements. A list of suggested polished wafer surface metal contamination inappropriate to circuits and devices is shown in Table 1 (based on SEMI M1). The inorganic contamination on silicon wafer surfaces is collected by VPD.

2.2 To quantify Ca and Fe, VPD/TXRF is used due to its sufficiently low detection limits. Na and Cu are quantified by VPD/GFAAS or VPD/ICP-MS. All analytical methods are widely used for the characterization of surface cleanliness.

2.3 This measurement technique can also be used to check the influence of certain process steps on minienvironments.

2.4 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.

Table 1 Suggested Polished Wafer Surface Metal Contamination Inappropriate to Circuits and Devices

Element	Test Method
Na	VPD/(AAS or ICP-MS)
Al	VPD/(AAS or ICP-MS)
K	VPD/(AAS or ICP-MS or TXRF)
Cr	VPD/(AAS or ICP-MS or TXRF)
Fe	VPD/(AAS or ICP-MS or TXRF)
Ni	VPD/(AAS or ICP-MS or TXRF)
Cu	VPD/(AAS or ICP-MS or TXRF)
Zn	VPD/(AAS or ICP-MS or TXRF)
Ca	VPD/(AAS or ICP-MS or TXRF)

3 Referenced Standards

3.1 SEMI Standards

SEMI C28 — Specifications and Guidelines for Hydrofluoric Acid

SEMI C35 — Specifications and Guidelines for Nitric Acid

SEMI E19 — Standard Mechanical Interface (SMIF)

SEMI M1 — Specification for Polished Monocrystalline Silicon Wafers

3.2 ISO Standards¹

ISO 9001 — Quality Systems—Model for Quality Assurance in Design, Development, Production, Installation, and Servicing

ISO 14644-1 — Cleanrooms and associated environments – Classification of air cleanliness

¹ International Organization for Standardization, ISO Central Secretariat, 1, rue de Varembe, Case postale 56, CH-1211 Geneva 20, Switzerland. Website: <http://www.iso.ch>

3.3 DIN Standards²

DIN 12650 Part 6 — Mechanical, physical and electrical laboratory apparatus; Piston operated volumetric apparatus; Gravimetric assessment of metrological reliability

NOTE 1: Unless otherwise indicated, all documents cited shall be the latest published versions.

4 Terminology

4.1 Abbreviations and Acronyms

4.1.1 *GFAAS* — Graphite Furnace Atomic Absorption Spectroscopy

4.1.2 *ICP-MS* — Inductively Coupled Plasma – Mass Spectroscopy

4.1.3 *PFA* — Perfluoroalkoxy

4.1.4 *PTFE* — Polytetrafluoroethylene

4.1.5 *PVDF* — Polyvinylidene fluoride

4.1.6 *TXRF* — Total Reflection X-Ray Fluorescence Spectroscopy

4.1.7 *ULSI* — Ultra Large Scale Integration

4.1.8 *VPD* — Vapor Phase Decomposition

4.2 Definitions

4.2.1 *box* — a protective portable container for a cassette and/or substrates.

4.2.2 *cassette* — an open structure that holds one or more substrates (e.g., wafer, masks).

4.2.3 *DI water* — deionized water (specified with specific resistivity $\geq 18 \text{ M}\Omega\text{cm}$, cations: Na, Cu, Fe, Ca $\leq 0.2 \text{ }\mu\text{g/L}$).

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

4.2.5 *pod* — a box having a Standard Mechanical Interface (SMIF) (See SEMI E19).

4.2.6 *reference wafer* — a cleaned wafer (see Section 8.2).

4.2.7 *sampling wafer* — a cleaned wafer (see Section 8.2), which will be or was exposed to the minienvironment for a certain time.

4.2.8 *standard mechanical interface (SMIF)* — the interface plane between a pod and another minienvironment (see SEMI E19).

4.2.9 *vapor phase decomposition* — a method in which impurities on the surface are collected by the so-called VPD procedure, i.e., the non-volatile products formed by acid decomposition of the oxide at the wafer surface are collected by a droplet of collecting agent, usually ultra-pure hydrofluoric acid or other reagent or combination of reagents, and the droplet subsequently being analyzed by AAS or ICP-MS, or dried in a manner which gives the least environmental contamination, the residue from the droplet subsequently being analyzed by TXRF.

5 Interferences

5.1 For worst cases, preconditioning of wafers can result in different surface properties indicated by different sensitivities for contamination absorption.

5.2 Non-linearity effects of the TXRF detector are significant at higher concentration levels ($> 10^{13}$ atoms/cm² under the detector area).

5.3 The collection efficiency of VPD depends on:

- the chemistry of the collecting solution
- the bonding of the metal impurities to the silicon surface
- the speed of the droplet, which is rolled over the wafer surface

5.3.1 Careful control of contamination and all other factors affecting the results such as solution concentrations, scanning methods and other procedures are necessary to obtain reproducible analytical results.

5.4 The measured TXRF intensity depends on the accuracy of the procedure to localize and to adjust to the sampled residue. It also depends on the distribution of different elements in and around the residue.

5.5 The detection of Fe or Ca using ICP-MS can be interfered with by background ions originated from the plasma unless some controlled measures are taken to minimize these interferences to acceptable levels.

6 Safety Precautions

6.1 Handling hydrofluoric acid is dangerous and shall be performed according to local regulations for laboratories. Operators shall be trained to deal with dangerous chemicals and vapors, especially hydrofluoric acid and HF vapor. Protective clothes and glasses must be worn when handling hydrofluoric acid.

7 Apparatus

7.1 The VPD treatment and contamination collection particularly, but also the handling and measurement of the specimen wafer is to be carried out in a specified

² Available from Deutsches Institut für Normung e.V., Beuth Verlag GmbH, Burggrafenstrasse 4-10, D-10787 Berlin, Germany. Website: <http://www.din.de>