

SEMI E45-1101 (Reapproved 0307)

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)

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NOTICE: This Standard or Safety Guideline has 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 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 mini-environments 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.

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

<i>Element</i>	<i>Test Method</i>
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)

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 Referenced Standards and Documents

3.1 SEMI Standards and Safety Guideline

SEMI C28 — Specifications for Hydrofluoric Acid

SEMI C35 — Specifications and Guide for Nitric Acid

SEMI E19 — Specification for 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

3.3 DIN Standard²

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.

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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 $\geq 18 \text{ M}\Omega\cdot\text{cm}$, 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).

¹ 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>

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

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

4.2.7 *sampling wafer* — a cleaned wafer (see § 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; that is, the nonvolatile 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.

5.3.2 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.

5.3.3 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 and controlled ambient (e.g., ISO Class 4 [as defined in ISO 14644-1]).

7.2 The VPD and the advisable drying chamber(s) shall have opening(s) made of PVDF, PFA, PTFE or similar resistant and pure polymer materials that are not attacked by HF. The chamber(s) may contain one or more wafer stacks. After evacuation, the chamber shall be flushed with filtered N₂ until the complete drying of the microdroplet residue is achieved.

7.3 For the aliquots of standard stock and scanning solutions, validated micropipettes shall be used. DIN 12650 Part 6 provides an applicable validation procedure.

8 Procedure

8.1 These procedures show the determination of Na, Ca, Cu, and Fe on a silicon wafer surface with VPD/GFAAS, VPD/ICP-MS and VPD/TXRF. For achieving best detection limits, VPD/GFAAS and VPD/ICP-MS shall be used for Na and Cu whereas VPD/TXRF shall be used for Ca and Fe.