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Standard Test Method for Oxidizable (Organic) Carbon on Wafer Surfaces (By Persulfate) [1]

This standard is issued under the fixed designation F 1228; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε1 NOTE—Keywords were added editorially in September 1994.

1. Scope

1.1 This test method covers wafers composed of silicon and other materials used in the semiconductor industry.

1.2 The test method is used to determine the mass of organic (oxidizable) carbon deposited on all exposed wafer surfaces. It is not used to determine total carbon content of the wafer material.

1.3 This test method is limited to carbon determinations in the range from 0.1 to 50 µg/sample. The detection limit is defined as 2 × standard deviation (2σ).

1.4 Volatiles may be lost during storage or handling, or both, of wafers. This test method is not used to quantify volatile organic compounds.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water [2]

D 1193 Specification for Reagent Water [2]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this test method, refer to Terminology D 1129.

4. Summary of Test Method

4.1 Carbon on wafer surfaces is determined by oxidation to carbon dioxide using a persulfate oxidant solution at 100 ± 1°C. The wafer is placed in a chamber filled with a clean, acidified persulfate solution and the heated chamber is sealed. Susceptible organic carbon is submitted to a 4 min digestion at 100°C to form carbon dioxide in solution. The resulting carbon dioxide is purged from solution and concentrated on a molecular sieve trap held at room temperature (25 to 28°C). The carbon dioxide is then thermally desorbed by heating the trap to 200 ± 10°C and is carried by nitrogen into a nondispersive infrared (NDIR) detector sensitized to carbon dioxide. The resulting detector signal (less the procedural blank) represents the mass of carbon oxide from the wafer surface. The carbon dioxide peak is quantized to carbon mass by either a linearized calibrated direct readout or comparison with signals resulting from oxidation of solutions of carbon standards.

5. Significance and Use

5.1 Organic carbon contaminants deposited on semiconductor wafers can cause losses in product yield. This test method makes possible a rapid quantification of surface contamination by carbon. Wafers can be tested before and after various stages of the wafer fabrication process. Results can be obtained within a few minutes of wafer sampling to aid in decisions concerning the wafer production process.

6. Interferences

6.1 Direct contact of a wafer with the human body or materials containing even small amounts of organic carbon compounds can cause a positive interference. Wafers must be handled with tools that have been cleaned of organic carbon.

6.2 Prolonged exposure of a clean wafer to the atmosphere can cause a positive interference due to adsorption of organics and carbon dioxide from the air or due to particle settling on the wafer. Wafers should be tested at time frame relevant to process.

6.3 Organic compounds are not oxidized at the same rates. Compounds may exist that are not completely oxidized in the standard time (4 min) allowed for reaction. In this case, a negative interference (loss of recovery) will result and if the slowly oxidizing compounds in question partition into the reaction solution, a positive interference (carry-over) may occur with the next sample. Thus, time of reaction must be demonstrated to be sufficient for complete oxidation of compounds present. Reaction time may be evaluated by analyzing a single wafer in replicate (without removing it from the chamber). If the result from the second carbon determination is greater than the blank by more than 0.1 µg, carbon was not sufficiently oxidized in the first determination. In this case,

[1] This test method is under the jurisdiction of ASTM Committee F-1 on Electronics and is the direct responsibility of Subcommittee F01.10 on Contamination Control Processing.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

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reaction time should be increased 1 min and the replicate test repeated with a new wafer until acceptable results are obtained.

6.4 Opening the digestion chamber introduces ambient carbon dioxide, that must be flushed from the chamber head space before each analysis. Carbon dioxide left in the head space will introduce a positive error in the results.

7. Apparatus

7.1 *Sealable Chamber*, suitable for maintaining a persulfate solution at 100 ± 1°C without corroding or producing carbon dioxide from the chamber materials is necessary. The chamber shall be of a configuration to accept wafers to be analyzed and must permit passage of nitrogen purge gas through the contained solution for purging of carbon dioxide (see Fig. 1). [3]

7.2 *Molecular Sieve Trap*—A trap containing 5Å molecular sieve, 30 to 60-mesh and a method of rapid trap heating capable of maintaining a trap temperature of 200 ± 10°C is required. The trap should rise from 25 to 200°C in less than 30 s. A stainless steel tube of 3.2-mm outside diameter and 2.7-mm inside diameter packed with 15 cm of 30 to 60-mesh molecular sieve is suitable as a trap (see Fig. 2).

7.3 *Non-Dispersive Infrared Detector*—A NDIR capable of detecting carbon dioxide in the range of 0.1 to 50 µg of carbon when this gas is carried through the detector at a carrier gas rate of 300 cm^3 min is needed.

7.4 *Valving*—A valving system capable of interfacing the digestion chamber, molecular sieve trap, and infrared analyzer, such that carbon dioxide from the digestion vessel can be purged from the chamber onto the trap, and that the carbon dioxide adsorbed on the trap can subsequently be desorbed while in-line with the detector is needed. The valving system should be designed to minimize contamination and handling of plumbing components. Fig. 3 shows a diagrammatic presentation of a suitable analyzer. [4]

7.5 *Tweezers*—A pair of clean TFE-fluorocarbon tweezers is needed to transfer wafers to and from the digestion chamber.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available. [5] Inferior grades may cause an increase in the procedural blank due to higher concentrations of carbon contaminants in the reagent.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D 1193. This water should contain less than 0.2 mg/L Total Oxidizable Carbon, (TOC).

8.3 *Acidified Sodium Persulfate (10 %) Solution*—Prepare by dissolving 100 ± 1 g of sodium persulfate ($Na_2S_2O_8$) into 1 L of reagent water. Add 5 mL of 85 % phosphoric acid (H_3PO_4). This solution can be stored in a closed borosilicate bottle at room temperature for 1 month.

8.4 *Gases*—Use nitrogen gas free of carbon dioxide and organic compounds. Zero grade nitrogen (99.99 %) and gases

[3] Digestion chamber such as a wafer toc, available from O. I. Corporation, P.O. Box 2980, College Station, TX 77841, or its equivalent, has been found suitable for this purpose.

[4] A suitable analyzer such as Wafertoc Model 700 TOC, or its equivalent, has been found suitable for this purpose.

[5] *Reagent Chemicals, American Chemical Society Specifications,* American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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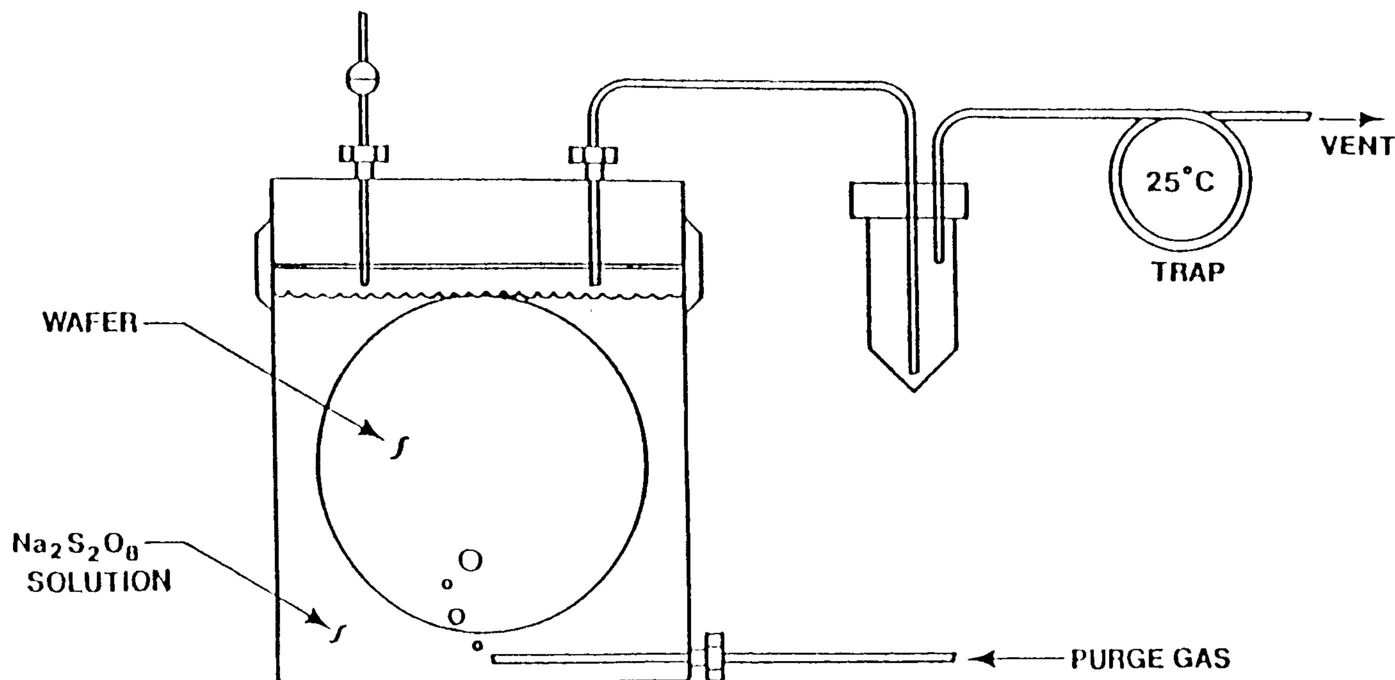

FIG. 1 Purging and Trapping CO_2 from Wafer Toc