NOTICE: This standard has either been superseded and replaced by a new version or withdrawn. Contact ASTM International (www.astm.org) for the latest information.

Designation: F 1239 – 94

AMERICAN SOCIETY FOR TESTING AND MATERIALS
100 Barr Harbor Dr., West Conshohocken, PA 19428
Reprinted from the Annual Book of ASTM Standards. Copyright ASTM

Standard Test Methods for Oxygen Precipitation Characterization of Silicon Wafers by Measurement of Interstitial Oxygen Reduction[1]

This standard is issued under the fixed designation F 1239; 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.

1. Scope

1.1 These test methods cover complementary procedures for testing the oxygen precipitation characteristics of silicon wafers. It is assumed that the precipitation characteristics are related to the amount of interstitial oxygen lost during specified thermal cycles.

1.2 These test methods may be used to compare qualitatively the precipitation characteristics of two or more groups of wafers.

1.3 These test methods may be applied to any *n*- or *p*-type, any orientation Czochralski silicon wafers whose thickness, resistivity, and surface finish are such as to permit the oxygen concentration to be determined by infrared absorption and whose oxygen concentration is such as to produce measurable oxygen loss.

1.4 These test methods are not suitable for determining the width or characteristics of a "denuded zone," a region near the surface of a wafer that is essentially free of oxide precipitates.

1.5 Because these test methods are destructive, suitable sampling techniques must be employed.

1.6 The values stated in SI units are regarded as standard.

1.7 *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.* Specific hazard statements are given in Section 8.

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification For Reagent Water [2]

F 416 Test Method for Detection of Oxidation Induced Defects in Polished Silicon Wafers [3]

F 612 Practice for Cleaning Surfaces of Polished Silicon Slices [4]

F 951 Test Method for Determination of Radial Interstitial Oxygen Variation [3]

F 1188 Test Method for Interstitial Atomic Oxygen Content of Silicon by Infrared Absorption [3]

2.2 SEMI Standards:

C 1 Specifications for Reagents [5]

C 3 Specifications for Gases [5]

2.3 Other Standard:

DIN 50 438, Part 1 Testing of Materials for Semiconductor Technology Determination of Impurity Content in Semiconductors by Infrared Absorption Oxygen in Silicon [3]

3. Summary of Test Method

3.1 A representative sample is selected from each group of wafers to be tested.

3.2 The initial value of interstitial oxygen concentration is measured by the infrared absorption method at the desired points on each wafer.

3.3 The wafers are passed through one of two simulation thermal cycles. Cycle A consists solely of a precipitation step. Cycle B consists of a nucleation step followed by a precipitation step.

3.4 After the thermal cycle, the oxide film is stripped and the final value of oxygen concentration is measured at the same points on each wafer using the same technique and instrumentation as was used to determine the initial value.

3.5 The oxygen reduction is determined for each wafer (or for each point on each wafer) tested as the difference between the initial and final values.

3.6 If all samples have the same initial oxygen concentration (within a narrow range), the average oxygen reduction for each test condition (such as, group or position on wafer) is computed, and the appropriate comparisons made.

3.7 If the samples have initial oxygen concentrations that cover a relatively wide range, a plot of oxygen reduction against initial oxygen concentration is made for each group or position. Again appropriate comparisons can be made.

4. Significance and Use

4.1 Oxide precipitates in the bulk of a silicon substrate wafer can act as gettering sites for contamination that may be

iTeh Standards (https://standards.iteh.ai) Document Preview ASTM F1239-94

[1] These test methods are under the jurisdiction of ASTM Committee F-01 on Electronics and are the direct responsibility of Subcommittee F01.06 on Silicon Materials and Process Control.

Current edition approved Aug. 15, 1994. Published October 1994. Originally published as F 1239 – 89. Last previous edition F 1239 – 89.

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

[3] *Annual Book of ASTM Standards*, Vol 10.05.

[4] Discontinued; see 1992 *Annual Book of ASTM Standards*, Vol 10.05.

[5] Available from Semiconductor Equipment and Materials International, 805 E. Middlefield Rd., Mountain View, CA 94043.

introduced during manufacture of circuits and devices. This contamination (usually metallic impurities) if not gettered, can reduce device manufacturing yields and degrade device or circuit performance. Thus, the oxygen precipitation characteristics of the substrate wafer can significantly affect both yields and performance.

4.2 Although interstitial oxygen concentration is the most important factor in affecting the amount of oxygen precipitation that occurs in silicon during a specific thermal cycle, the presence of other impurities such as carbon and differences in dopant type and density, thermal history, or defect properties of the crystal can also affect the precipitation characteristics. Thus, it is frequently necessary to choose particular characteristics for a particular application.

4.3 These test methods may be used to compare the oxygen precipitation characteristics of two or more groups of silicon wafers. These test methods are based on thermal cycles that simulate certain common device processing cycles.

4.3.1 *Cycle A*, a one-step precipitation cycle, provides an indication of the native nucleation sites present in the as-received wafers.

4.3.2 *Cycle B*, a two-step nucleation-precipitation cycle, simulates the precipitation that occurs in normal *n*-MOS device processing.

4.4 These test methods may also be used to determine the uniformity of oxygen precipitation characteristics across a wafer or from wafer to wafer within a lot.

4.5 Determination of material performance in actual device fabrication situations is beyond the scope of these methods. However, by comparing the results of these tests with actual device yields and performance, criteria for selection of specific material characteristics may be established.

5. Interferences

5.1 All factors that affect the infrared absorption measurement (including differences in back surface condition, instrumental characteristics, and wafer resistivity) may cause errors in the determination of oxygen reduction.

5.2 If significant quantities of oxygen are outdiffused during the thermal cycles, the measured oxygen reduction may not be representative of the amount of oxygen precipitation.

5.3 If precipitate size varies from sample to sample, the variations in measured oxygen reduction may not be representative of variations in the number of oxide precipitates that are formed.

5.4 The specified thermal cycles may or may not provide adequate simulation of the cycles used in a particular device processing sequence. The results obtained in these test methods can serve as predictors of those expected in actual device processing only to the extent that the simulation is representative of the device process.

6. Apparatus

6.1 *Infrared Absorption Spectrophotometer*, as specified in Test Method F 1188 or DIN 50 438, Part 1.

6.2 *Resistance Heated Tube Furnace*, capable of providing temperatures in the range from 750 to 1050°C to ±2°C over the length required to contain the load of wafers to be tested. The furnace shall be fitted with the following:

6.2.1 *Gas Manifold*, that allows dry oxygen and nitrogen to be mixed at the required ratios and flows, (see Table 1);

6.2.2 *Quartz, Polysilicon, or Silicon Carbide Tube*, of diameter appropriate for the wafers to be tested to isolate the wafers from external contamination;

6.2.3 *Quartz Boats*, to hold the wafers during processing;

6.2.4 *Loader*, to allow controlled insertion of the quartz boat into the hot zone, (see Table 1); and

6.2.5 *Laminar Flow Load Station*, to permit loading of the wafers without adding contamination to the surfaces.

6.3 Facilities for processing wafers through chem-mechanical polishing, or bright acid etching to provide smooth, flat surfaces.

6.4 Facilities for dipping the wafers in hydrofluoric acid prior to the oxygen determination in order to remove the surface oxide film grown during thermal cycling. Suitable protective clothing, acid disposal facilities, and ventilation shall be provided.

6.5 Facilities for cleaning wafers by a standard process, for example, that specified in Practice F 612.

6.6 *Scribe or Laser Marker*, for marking the wafers with unique identification (unless premarked wafers are available).

7. Reagents and Materials

7.1 *Electronic Grade Hydrofluoric Acid*, in accordance with SEMI Specification C 1.8.

7.2 *Electronic Grade Oxygen*, in accordance with SEMI Specification C 3.16.

7.3 *Carrier Grade Nitrogen*, in accordance with SEMI Specification C 3.15.

7.4 *Deionized Water*, with a resistivity equal to or greater than that specified for Type II Reagent Water in Specification D 1193.

iTeh Standards (https://standards.iteh.ai) Document Preview
ASTM F1239-94
https://standards.iteh.ai/catalog/standards/sist/b351480d-979f-4d95-9dc6-212d82f5863a/astm-f1239-94

8. Hazards

8.1 The acids used in these test methods are hazardous. All precautions normally used with these chemicals should be strictly observed. Obtain and read the material safety data sheet prior to use of any chemical.

9. Selection and Preparation of Test Specimens

9.1 Choose test wafers from each group being tested in such a way as to cover the entire range of oxygen concentration found in the group. Choose at least two wafers with oxygen concentration in each 0.5 ppm (IOC-88)[6] interval in the range.

[6] As defined in Test Method F 1188.

TABLE 1 Thermal Cycle Tests for Oxygen Precipitation in Silicon

Test A	1050°C for 16 h
Test B	750°C for 4 h followed by 1050°C for 16 h
Furnace Ambient	Nitrogen plus 5 % dry oxygen
Gas Flow Rate	4.2 ± 0.2 L/min [A]
Push/Pull Temperature	750°C
Push/Pull Rate	25 cm/min
Ramp Up Rate	10°C/min
Ramp Down Rate	5°C/min

[A] For 155 mm diameter tube; for other diameters flow rate should be proportional to the cross sectional area of the tube.