



Designation: F 1227 – 89 (Reapproved 1999)

Standard Test Method for Total Mass Loss of Materials and Condensation of Outgassed Volatiles on Microelectronics-Related Substrates¹

This standard is issued under the fixed designation F 1227; 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 This test method covers various thermoplastics and thermosets routinely used in the microelectronics industry. This test method covers a screening technique to determine volatile content of these materials when exposed to a vacuum environment. Two parameters are measured: total mass loss (TML) and collected volatile condensable materials (CVCM).

1.2 This test method describes the test apparatus and related operating procedures for evaluating the mass loss of materials being subjected to 25, 70, or 125°C at less than 5 Pa (0.05 mbars) for 24 h. The lowest temperature simulates storage conditions, while the middle and upper temperatures simulate soft bake and hard bake conditions encountered during microelectronics manufacturing. The overall mass loss can be classified into noncondensables and condensables. The latter are characterized herein as being capable of condensing on a silicon wafer, glass plate, aluminum disk or other substrate, maintained at a temperature of 18°C.

NOTE 1—Unless otherwise noted, the tolerances are 18°C \pm 3°C, 25°C \pm 1°C, 70°C \pm 1°C, and 125°C \pm 1°C.

1.3 Any thermoplastic or thermoset can be tested. The materials may be tested in the “as-received” condition or prepared for test after various curing or manufacturing steps.

1.4 This test method is primarily a screening technique for materials and is not necessarily valid for computing actual contamination on a system or component because of possible differences in configuration, temperatures, and material processing. The three temperatures allow testing with respect to maximum expected service temperature. Also, many polymers cannot be tested at 125°C as they melt or undergo glass transition above 70°C.

1.5 The use of materials that are deemed acceptable in accordance with this test method does not ensure that the system or component will remain uncontaminated. Therefore, subsequent functional, developmental, and qualification tests should be used, as necessary, to ensure that material performance is satisfactory.

¹ This test method is under the jurisdiction of ASTM Committee E-21 on Space Simulation and Applications of Space Technology and is the direct responsibility of Subcommittee E21.05 on Contamination.

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1.6 No data is yet available to evaluate minimum sample sizes. Sample sizes as small as 130 mg have been used.

1.7 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.8 *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:

E 595 Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment²

3. Terminology

3.1 Definitions:

3.1.1 *collected volatile condensable material (CVCM)*—the quantity of outgassed matter from a test specimen that condenses on a silicon wafer maintained at a specific constant temperature for a specified time. CVCM is expressed as a percentage of the initial specimen mass and is calculated from the condensate mass determined from the difference in mass of the silicon wafer before and after the test.

3.1.2 *total mass loss (TML)*—total mass of material outgassed from a specimen that is maintained at a specified constant temperature and operating pressure for a specified time. TML is calculated from the mass of the specimen as measured before and after the test and is expressed as a percentage of the initial specimen mass.

4. Summary of Test Method

4.1 The test specimen may be a raw material or cut from a device. It may be preferable to obtain samples from completed devices, so that any volatiles that are released during the manufacturing process are not included in TML. A specimen is added to a clean, preweighed boat and boat and specimen are weighed. The loaded boat is then inserted into a sample chamber in the copper heating bar. Up to 16 specimens can be

² *Annual Book of ASTM Standards*, Vol 15.03.

tested simultaneously in the apparatus. The sample chambers are covered and the apparatus is sealed with a bell jar and evacuated to a vacuum of at least 5 Pa (0.05 mbars). Test temperatures are chosen according to expected normal use temperatures of the product and the melt or glass transition temperature of the material. The raised temperature causes vapor from the specimen to travel through a hole in the sample chamber to the collector chamber. Some of the vapor may condense on a preweighed silicon wafer (or other substrate) that is maintained at 18°C. Each chamber is isolated from the others to prevent cross contamination. After 24 h, the system is cooled and repressurized with room air. The specimen and wafers are weighed. From these results and the specimen and wafer weights determined previously, the percentage TML and percentage CVCM are obtained. Fourteen samples are generally averaged (two sample chambers contain controls) to obtain reported values.

NOTE 2—The condensate on the collector plates may be analyzed by various techniques such as infrared spectrophotometry in accordance with Test Method E 595, Note 2.

4.2 One randomly selected sample chamber in each heating bar is used for an empty weighed boat. These boats act as controls to monitor system background error and to detect inadequate cleaning procedures.

5. Significance and Use

5.1 This test evaluates the changes in the mass of a test specimen on exposure under vacuum to temperatures of 25, 70, and 125°C, and the mass of those products that leave the specimen and condense on a silicon wafer at a temperature of 18°C.

5.2 Outgassing properties may be compared only with properties obtained at the same temperature. Glass plates or other substrates are acceptable substitutes for the silicon wafers. However, CVCM results obtained using alternate collector plates are only comparable with others sharing similar collector plate composition and geometry. Therefore, the type of collector should be indicated in the report.

5.3 Use of a vacuum, while not always representative of actual use conditions, accelerates the outgassing process and prevents contamination from background gases.

5.4 Some materials may be eliminated at 125°C during the outgassing screening process because of unsatisfactory TML or CVCM. However, their properties may prove acceptable at 70 or 25°C. Materials that condense only below 18°C will not be detected.

5.5 Samples shall be stored in airtight containers when received and are removed from these only minutes before weighing. This controls contamination from handling and minimizes the effects of environmental changes in humidity. Samples are weighed at room temperature.

6. Apparatus

6.1 *Electronic Analytical Microbalance*, with 0.1- μ g readability.

6.2 *Two Copper Heating Bars*, 241.3 mm (9.50 in.) in length, 25.4 mm (1.00 in.) thick and 88.9 mm (3.50 in.) in width, are used in the outgassing apparatus. Each bar contains eight cylindrical sample chambers, 25.4 mm (1.00 in.) in

diameter. A hole to the rear of each sample chamber allows gases from the sample to pass into a collector chamber, where a 38.1-mm (1.50-in.) silicon wafer functions as a collector plate.³ The collector chamber and wafer are maintained at 18°C \pm 3°C via a water-cooled aluminum plate (see Figs. 1-4 for a schematic diagram and critical dimensions).

6.3 The test apparatus is mounted on the base plate of a vacuum system within a 308-mm (12.125-in.) diameter vacuum bell jar. The operation of the vacuum system is automatically controlled. Power to the heating element mounted in the copper bars is regulated through temperature controllers.

7. Test Specimen

7.1 Cylindrical aluminum sample boats, 6.3 mm (0.25 in.) in depth and 15.2 mm (0.60 in.) in diameter are used (see Fig. 5).

7.2 Normally, raw plastic pellets are tested in the “as-received” condition. Large pellets and other materials such as finished parts, films, and tubing, are cut with a clean tool into pieces small enough to fit into the boats.

7.3 The sample materials must not be contaminated or handled with bare hands before testing. Human skin oils may result in inaccurate TML and CVCM results. Therefore, degreased metal tweezers or forceps are required during sample preparation.

8. Procedure

8.1 Before operation, the sample chambers are wiped with acetone. Sixteen boats and silicon wafers are soaked in a suitable solvent for 1/2 h, then drained and dried under a hood for an additional 1/2 h. They are stored in a desiccator for 12 to 24 h before use. Calibrate the balance. Record all weights to 0.1 μ g.

8.2 Weigh each silicon wafer and record the weights. Mount each wafer in its cooling chamber receptacle.

8.3 Weigh each aluminum boat and record the weights on the Outgassing Experimental Data sheets (Fig. 3). Add 100 to 300 mg of sample to each boat and reweigh the boats and samples. Record the weights.

8.4 Place each boat with sample into a sample chamber.

8.5 Mount and screw down the cover plates onto the entry end of the sample chambers.

8.6 Cover the vacuum system with the bell jar and evacuate it to 5 Pa (0.05 mbars) within 1/2 h. Turn on the heater controls and adjust the controllers to one of the three recommended temperatures (25, 70, or 125°C). Maintain the collector plate temperature at 18°C.

8.7 Let the system operate for 24 h, then open the gas ballast to the vacuum system and turn off the temperature controllers. Remove the bell jar to allow the system to return to room atmospheric conditions.

8.8 Remove wafers from their cooling receptacles and weigh immediately.

³ Silicon wafers, 38.1 mm (1.5 in.) in size, available from the Unisil Corp., 405 National Ave., Mountain View, CA 94043, or equivalents, have been found suitable for this purpose.

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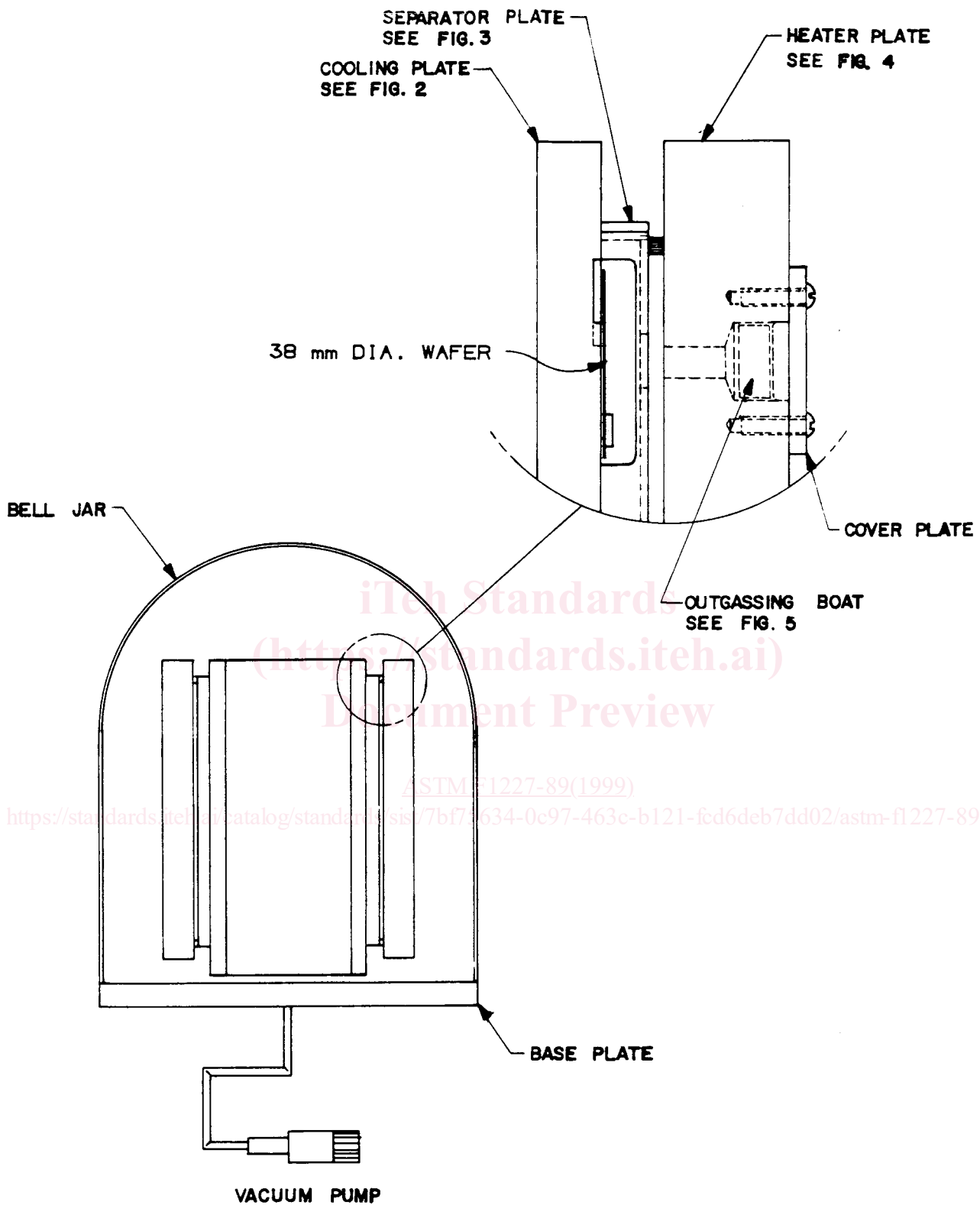


FIG. 1 Schematic of Outgassing Apparatus

8.9 Remove boats with samples from the sample chambers. When cool, reweigh.

$$\% \text{ TML} = (SB_F - SB_I) / S_I \times 100$$

(1)

9. Calculation

9.1 Calculate % TML as follows: