Designation: F 542 – 02

An American National Standard

Standard Test Method for Exothermic Temperature of Encapsulating Compounds for Electronic and Microelectronic Encapsulation[1]

This standard is issued under the fixed designation F 542; 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 provides results that are related to the maximum temperature reached in a specific volume by a reacting liquid encapsulating compound, and the time from initial mixing to the time when this peak exothermic temperature is reached.

1.2 This test method provides a means to measure the peak exothermic temperature of an encapsulating compound.

1.3 *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.* For specific hazard statements see Section 8.

NOTE 1—There is no equivalent IEC standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1711 Terminology Relating to Electrical Insulation[2]

D 5423 Specification for Forced-Convection Laboratory Ovens for Evaluation of Electrical Insulation[3]

3. Terminology

3.1 *Definitions:* For definitions of terms used in this test method, refer to Terminology D 1711.

3.2 *Definition of Term Specific to This Standard:*

3.2.1 *encapsulating compound, n*—a resin system used to encase electronic components.

3.2.1.1 *Discussion*—These resins are generally used to provide protection from the operating environment and mechanical damage.

4. Summary of Test Method

4.1 A thermocouple is used to measure the highest temperature at the geometric center of a volume of encapsulating compound in order to characterize the heat generated by the chemical exothermic reaction.

5. Significance and Use

5.1 Heat generated by a reacting liquid encapsulating compound may cause damage to heat-sensitive electronic components. Degradation of the encapsulating compound may also occur at high temperatures. Proper selection of an encapsulate must include knowledge of its exothermic temperature to preclude damage to components.

5.2 Since the exothermic temperature of a reacting encapsulating compound varies with the volume and geometry of material, it is essential that the volume and geometry be specified in any determination. By selection of an appropriate volume and geometry, the exothermic temperature may be measured in sufficiently precise and reproducible form for application evaluation, quality control, and encapsulating compound characterization.

5.3 Materials may be compared by testing equal volumes of each material using the same geometry. Two different volumes of the same material in similar geometries may be tested to determine the effect of volume on the exothermic temperature.

6. Interferences

6.1 Under normal electronic system encapsulation, the heat sink of the components would reduce the maximum heat generated in the encapsulating compound. Since the volume and geometry of the electronic system, plus the heat sink of the components, affect the exothermic temperature of the encapsulating compound, it is recommended that actual hardware be tested in cases in which temperature-sensitive electronics are utilized.

7. Apparatus

7.1 *Specimen Containers* for specific test volumes, as follows:

7.1.1 For 50-mL test, borosilicate glass beaker, Griffin,

---

[1] This test method is under the jurisdiction of ASTM Committee D09 on Electrical and Electronic Insulating Materials, and is the direct responsibility of Subcommittee D 09.01 on Electrical Insulating Varnishes, Powders and Encapsulating Compounds.

Current edition approved Sept. 10, 2002. Published October 2002. Originally published as F 542 – 77 T. Last previous edition F 542 – 98.

[2] *Annual Book of ASTM Standards,* Vol 10.01.

[3] *Annual Book of ASTM Standards,* Vol 10.02.

7.1.2 For 125-mL test, "No. 202 × 204" standard canning industry 130 mL can, 54.0 mm in diameter by 57.1 mm in height,

7.1.3 For 150-mL test, borosilicate glass beaker, Griffin, low-form,

7.1.4 For 200-mL test, "No. 202 × 314" standard canning industry 225 mL can, 54.0 mm in diameter by 98.4 mm in height, or

7.1.5 For 300-mL test, "No. 211 × 400" standard canning industry No. 1 "soup" can, 68.2 mm in diameter by 101.6 mm in height.

7.2 *Glass Tubing*, of sufficient length and diameter to support the thermocouple wire into the geometric center of the specimen container.

7.3 *Thermocouple*, expendable, one for each specimen, capable of measuring temperatures from 23 to 260°C (73 to 500°F), inclusive.

7.4 *Temperature-Indicating Device*, utilizing thermocouples, and accurate to ± 3°C (± 5°F) (see Note 2).

NOTE 2—Automatic temperature recorders have been used successfully.

7.5 *Balance*, capable of measuring the mass of the compound ingredients with accuracy of ±1 %.

7.6 *Oven*, forced-air circulating type, conforming to Type II of Specification D 5423.

8. Safety Precautions

8.1 Some encapsulating compounds exhibit exothermic reactions, to the point of boiling or autoignition in the center of the compound mass, and possible explosive reactions can occur. For unknown compounds, run the test with the smallest volume first.

8.2 Shrinkage of the encapsulating compound during the curing process can cause fracture of the glass containers if bonding to the glass occurs. Use suitable mold release compounds on all glass specimen containers.

8.3 Special handling precautions are required because some encapsulating compounds are known to contain toxic components. Follow the manufacturer's precautionary instructions and sound laboratory safety practices.

8.4 The use of face shields or other suitable protection may be necessary when testing is conducted in glass containers.

9. Sampling

9.1 Because of the diverse nature of the encapsulating compounds, and the various commercially available forms and packages of resins, hardeners, catalysts, etc., no standard methods of sampling have been established. Select an adequate amount of material, which contains each ingredient, to permit the preparation of the specimens required.

10. Conditioning

10.1 Unless otherwise specified, condition the compound ingredients, usually a resin and a hardener, at 23 ± 2°C for room-temperature systems or at the manufacturer's specified preheating temperature for oven-curing systems. Record the time and temperature of the conditioning.

11. Procedure

11.1 Select one or more volumes suitable to the end use. Test three specimens for each volume selected.

11.2 Position a thermocouple into the approximate geometric center of each specimen container by means of the glass tubing as shown in Fig. 1. Hold the glass tubing in place by a clamp situated above the specimen container in a manner such that the thermocouple extends below the glass tubing and is in a position to be wetted by the encapsulating compound.

11.3 Determine the mass of the compound ingredients in accordance with the manufacturer's directions and mixing ratios. Dispense and weigh sufficient material to ensure an amount to fill the specimen holder.

11.4 Mix the compound ingredients in accordance with the manufacturer's directions. Record as zero time the time at which the curing agent or hardener is added to the base resin and mixing is begun.

NOTE 3—Vacuum degassing is recommended provided it is consistent with the chemistry of the system.

11.5 Within 5 min from zero time pour the mixed compound into each specimen container to approximately 3 mm from the top of the container (see Fig. 1). Take precautions during pouring to avoid entrapping air. After pouring, make sure the glass tube containing the thermocouple is located in the center of the specimen container.

11.6 Cure the poured specimens as agreed upon by the parties to the test. Record the curing temperatures and times as the cure schedule. Cure heat-cured compounds in a forced-air circulating oven. Cure room temperature systems at 23 ± 2°C.

11.7 Monitor the thermocouple readings and record the maximum temperature and the time at which it occurs to the nearest 1 minute. These are the peak exothermic temperature and peak exothermic time. The peak exothermic temperature has been reached when a decline in temperature is observed for 10 min or longer.

11.8 Record any observations in regard to the condition of the cured specimen, such as cracking, shrinkage, or charring.

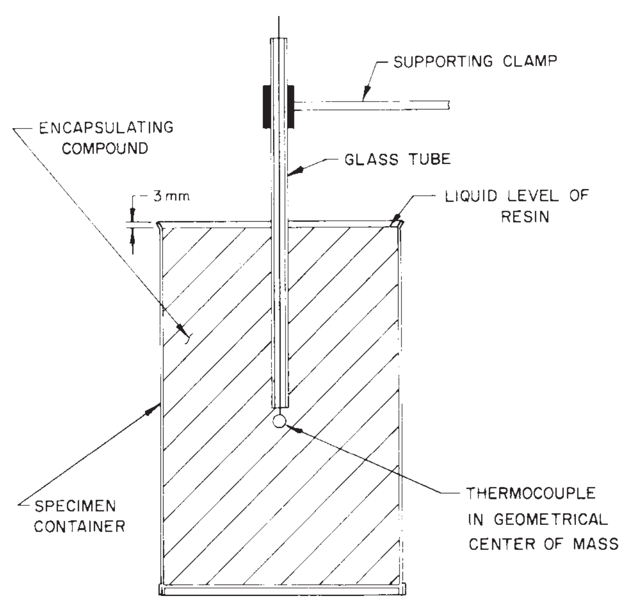

FIG. 1 Test Specimen for Exothermic Temperature

11.9 Compute the average peak exothermic temperature and time for each volume tested.

12. Report

12.1 Report the following information:

12.1.1 Date of test,

12.1.2 Identification of encapsulating compound under test, mass of each component for each mixing, time and temperature of conditioning, and the cure schedule,

12.1.3 Test volume, mL,

12.1.4 Number of test specimens,

12.1.5 Peak exothermic temperature, °C, for each specimen,

12.1.6 Average peak exothermic temperature, °C, for each specimen volume for each compound tested,

12.1.7 Peak exothermic time, min., for each specimen,

12.1.8 Average peak exothermic time, min., for each specimen volume for each compound tested, and

12.1.9 Any observations in regard to the condition of the cured specimen, such as cracking, shrinkage, or charring.

13. Precision and Bias

13.1 *Precision*:

13.1.1 An interlaboratory study was conducted in which eight laboratories tested three encapsulating compounds in two test volumes for each compound.

13.1.2 The coefficient of variation for the peak exotherm within laboratories was 2.67 %, and between laboratories was 7.56 %.

13.1.3 The coefficient of variation for the peak exothermic time within laboratories was 6.00 %, and between laboratories was 12.73 %.

13.2 *Bias*—This method has no bias, as the values for peak exothermic temperature and peak exothermic time are defined solely in terms of this method.

14. Keywords

14.1 electronic; encapsulating compounds; exothermic temperature; microelectronic encapsulation