Designation: D3532/D3532M – 12

Standard Test Method for Gel Time of Carbon Fiber-Epoxy Prepreg[1]

This standard is issued under the fixed designation D3532/D3532M; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the determination of gel time of carbon fiber-epoxy tape and sheet. The test method is suitable for the measurement of gel time of resin systems having either high or low viscosity.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

1.2.1 Within the text, inch-pound units are shown in brackets.

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.*

2. Summary of Test Method

2.1 A specimen of prepreg material approximately 5 mm [0.25 in.] square is placed between microscope cover glasses on a hot plate preheated to either of two test temperatures, 120 or 175°C [250 or 350°F]. Pressure is applied to the specimen through the coverglass with a wood pick to form a bead of resin at the edge of the cover glass. The time from the application of heat until the resin ceases to form strings by contact with the pick is noted and reported as the gel time.

3. Significance and Use

3.1 This test method can be used to obtain the gel time of resin squeezed from prepreg tape or sheet material. This test is a useful measure for material acceptance.

3.2 The gel time will vary with the test temperature. The temperatures specified in this test method are two of many temperatures often used in processing epoxy prepreg material. If other test temperatures are used, this is to be clearly noted as indicated in 9.1.2.

3.3 Gel time is not recommended as a measure of outline (unacceptable degree of cross-linking). Use Resin Flow Test Method D3532, or Dynamic Viscosity Practice D4473.

4. Apparatus

4.1 *Cutting Knife.*

4.2 *Hot Plate,* capable of maintaining temperatures of either 120°C [250°F] or 175°C [350°F] and the means of measuring its surface temperature to an accuracy of ±1°C [±2°F].

4.3 *Stopwatch or Suitable Timer,* capable of reading 1-s intervals up to 60 min.

4.4 *Microscope Coverglasses,* 18 to 22 mm [0.7 to 0.9 in.] in diameter.

4.5 *Wooden Probe,* small, such as a toothpick, mounted in a drafting pencil holder or fine glass rod. A low thermal capacity is needed.

5. Interferences

5.1 The test is a subjective measure of when a gel point is reached. The visual evidence of gel may vary between materials, reinforcements, and, in some cases, between resin material batches. If the definition of gel in this method is not sufficiently standard for a given material, interested parties shall agree on a further definition of gelation.

6. Test Specimen

6.1 A minimum of three specimens shall be tested for each sample.

6.2 The test specimen shall consist of prepreg material cut to approximately 6-mm [0.25-in.] square.

7. Conditioning

7.1 Store carbon fiber-epoxy prepreg tape at low temperatures, approximately −18°C [0°F], to prolong the usefulness of the material. Allow the sealed packages of material to warm to ambient temperature before the seal is opened to ensure that the material does not absorb moisture from the atmosphere.

[1] This test method is under the jurisdiction of ASTM Committee D30 on Composite Materials and is the direct responsibility of Subcommittee D30.03 on Constituent/Precursor Properties.

Current edition approved May 1, 2012. Published June 2012. Originally approved in 1976. Last previous edition approved in 2009 as D3532 – 99(2009). DOI: 10.1520/D3532_D3532M-12.

8. Procedure

8.1 Cut the specimen of prepreg.

8.2 Preheat the hot plate to either test temperature A, 120 ± 1°C [250 ± 2°F] or B, 175 ± 1°C [350 ± 2°F] or the temperature that has been specified.

8.3 Place a cover glass on the hot plate and allow 20 s for it to reach temperature.

8.4 Place the specimen in the center of the coverglass and cover it with another coverglass.

8.5 Commence timing immediately.

8.6 Probe the surface of the coverglass to apply pressure to the prepreg specimen. Use the probe to isolate a bead of resin at the edge of the specimen. Continue to probe the bead of resin and observe its tendency to form strings. Select the point at which strings cannot be formed by contact with a pick as the gel time. Record this time as gel time.

9. Report

9.1 The report shall include the following.

9.1.1 Complete identification of the material including the fiber type, the fiber manufacturer, surface treatment of the fiber, the resin type, the resin manufacturer, and prepreg supplier,

9.1.2 The test temperature, and

9.1.3 The gel time reported to the nearest 1 s.

10. Precision and Bias

10.1 The precision, defined as the degree of mutual agreement between individual measurements, can be estimated from the results of a round robin conducted on two samples of prepreg by four laboratories, each laboratory making three measurements of each sample. The coefficient of variation for measurements on one material, mean gel time of 6.8 min was 41.7 % and 51.6 % on the other material which had a mean gel time of 5.6 min. No modern precision statement is available.

10.2 No estimate of bias can be offered as no accepted reference level is available.

11. Keywords

11.1 carbon fiber-epoxy prepreg; gel time