Designation: D 3531 – 99

Standard Test Method for Resin Flow of Carbon Fiber-Epoxy Prepreg[1]

This standard is issued under the fixed designation D 3531; 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 the amount of resin flow that will take place from prepreg tape or sheet under given conditions of temperature and pressure.

1.2 The values stated in SI units are to be regarded as standard. The values in parentheses are for reference only.

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 weighed specimen consisting of two plies a minimum size of 50-mm (2.0-in.) square 0–1.57 rad (0 to 90°) crossplied tape is sandwiched between bleeder material and release film. The sandwich is placed in a platen press heated to either temperature A, 120°C (250°F), or temperature B, 175°C (350°F) or any other temperature specified. The press is closed to provide a pressure of 700 kPa (100 psi). The pressure is held for 15 min or until the resin gels. The cooled sandwich assembly is removed and the resin that has flowed to the edges of the specimen is removed and the specimen reweighed. The change in weight is expressed as a percent of the original weight and reported as percent flow.

3. Significance and Use

3.1 This test method is used to obtain the resin flow of carbon fiber-epoxy prepreg tape or sheet material. It is suitable for comparing lots of material of supposedly the same characteristics and also for comparative evaluation of materials produced by different vendors using different resin-fiber combinations.

3.2 Composite parts are laminated from prepreg material at various pressures and temperatures. Production process design will require a flow test be run at a temperature and a pressure close to that of the actual molding conditions. All methods of measuring resin flow are dependent on the size and geometry of the specimen. This test method uses the smallest quantity of tape that will give reproducible results.

3.2.1 The percent resin flow of a single fiber and resin system at a temperature and pressure varies with the volatile content, degree of advancement of epoxy resin, and with the resin content of the prepreg tape or sheet.

3.2.2 As volatile content and degree of resin cure (advancement) change with time, this test is useful in comparing the life of prepreg tape and sheet.

4. Apparatus

4.1 *Cutting Template*, square metal, 50 by 50 mm (2.0 by 2.0 in.), minimum.

4.2 *Cutting Template*, metal, 100 by 100 mm (4.0 by 4.0 in.), minimum.

4.3 *Cutting Knife*, single edge.

4.4 *Analytical Balance* capable of weighing to the nearest 0.001 g.

4.5 *Glass Bleeder Cloth*, Style 1581 or 181.

4.6 *TFE-Fluorocarbon Coated, Woven Separator Cloth,*[2] porous.

4.7 *Release Film* of 0.03 to 0.06 mm (0.001 to 0.002 in.) thickness polyester, aluminum, etc.

4.8 *Platen Press*, capable of being heated to 175 ± 3°C (350 ± 5°F) and capable of applying 4000 N (900 lbf).

5. Interferences

5.1 This method depends on platen force being supplied evenly to the specimen. For this to be done, the platen must load evenly across its surface and not point load to the point of initial contact. When bleeder materials are used on the top and bottom of the specimen, the effect of uneven pressure application is less pronounced than if no bleeder materials are used. Bleeders tend to minimize pressure effects, since if resin flows into the bleeder it will do so within a broad pressure range. Sometimes, platen pressure needs to be increased gradually to assure even loading.

5.2 The platen flatness must be sufficient for the specimen to load evenly. For this reason the specimen thickness should be at least five times the tolerance of platen flatness. Specimens that do not meet this requirement should have additional ply layers oriented as a repeating unit of the first two plies.

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[1] This test method is under the jurisdiction of ASTM Committee D-30 on Composite Materials and is the direct responsibility of Subcommittee D30.03 on Constituent/Precursor Properties.

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[2] DuPont product TX-1040 or equivalent has been found satisfactory for this purpose.

5.3 Ply orientation and coupon size directly affects reported flow. A sample cut with a ply orientation of 0.78 radians (45°) will not have the same reported flow as a sample cut with a ply orientation of 0 radian (0°). This is because flow paths are hindered to a different degree based on the different coupon size and orientation.

5.4 Temperature should be even across the specimen and within the tolerance specified. Temperature influences resin viscosity, which effects flow rate.

5.5 Generally, larger coupon sizes reduce lateral flow since resin has further to travel to the edge of the specimen. Larger coupon sizes do not greatly influence horizontal flow (with bleeders). However, difficulty in coupon handleability increases with increasing coupon size. Also small coupon size of 50 mm (2.0 in.) square is felt to be the minimum coupon size. A maximum practical size is 100 mm (4.0 in) square.

5.6 The matching of angles and footprint from one ply to the next is critical. Flow differences may be found if the ply layers do not superimpose on top of each other or are aligned with an angle bias.

5.7 It is recommended that the heated platens remain closed under pressure until resin gelation occurs. Shorter times may cause some of the flowed resin to associate back with the sample rather than the bleeder cloth. Leaving the sample in the press after gelation has no effect on flow results.

6. Test Specimen

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

6.2 The test specimen shall consist of two plies, one at 0 rad (0°) and the other at 1.57 rad (90°) of 50-mm (2-in.) square minimum size prepreg sheet.

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.

7.2 Do not expose the material, which usually has some volatile content, at ambient temperature for long periods of time before testing is begun.

8. Procedure

8.1 Cut two pieces 50-mm minimum ±1 mm (2.0-in. ± 0.04 in.) square from the prepreg material. Other sizes may be used, but the two plies shall match within the tolerences specified.

8.2 Crossply the pieces at 0 to 1.57 rad (0 to 90°) and weigh to nearest 0.001 g, recording the weight as W_1.

8.3 Cut four pieces of Style 1581 glass bleeder cloth of at least four times the area of the prepeg sample, 100 by 100 mm (4.0 by 4.0 in.) square, minimum.

8.4 Cut two pieces of porous TFE-fluorocarbon separator cloth to the same size as the bleeder cloth, 100 by 100 mm (4.0 by 4.0 in.) square, minimum.

8.5 Cut two pieces of release film minimum of 150 mm (6.0 in.) square.

8.6 Prepare the specimen assembly as follows:

8.6.1 Place a 150- by 150-mm (6.0- by 6.0-in.) square piece of release film on a clean work surface.

8.6.2 Apply two pieces of bleeder cloth centered upon the 150-mm (6.0-in.) square of release film.

8.6.3 Place one piece of porous separator cloth on top of and with the edges aligned to the edges of the glass bleeder cloth.

8.6.4 Place the 50-mm (2.0-in.) square test specimen on the center and parallel with the edges of the porous separator cloth.

8.6.5 Cover the specimen with another piece of porous separator cloth.

8.6.6 Cover the assembly with two pieces of the bleeder cloth aligned with the edges.

8.6.7 Complete the assembly by covering it with a 150-mm (6.0-in.) square piece of release film. All edges should be aligned and centered on the sheets.

8.6.8 Weigh the specimen assembly to nearest 0.001 g and record as W_2.

8.6.9 Insert the assembly into a platen press preheated to either of the test temperatures (A or B) ±3°C (±5°F) or any other temperature specified. Record the actual platen temperatures. Cure this assembly for the gel time recommended by the material supplier. Apply pressure of 700 (±70) kPa (100 ± 10 psi) within 5 s of closing platens and starting timer. Cure this assembly for the gel time recommended by the material supplier.

8.6.10 Remove the layup assembly from the press.

8.6.11 Allow assembly to cool to room temperature, reweigh to nearest 0.001 g, record as W_3.

8.6.12 Separate the resin saturated bleeder materials from the composite specimen. Use care in separating the separator material and the specimen to avoid loss of fiber material.

8.6.13 Reweigh the specimen to the nearest 0.001 g and record as W_4.

9. Calculation

9.1 Calculate the resin flow, RF, as a weight percent of the original prepreg specimen as follows:

$$RF, \% = \frac{W_1 - W_4}{W_1} \times 100 \quad (1)$$

where:
W_1 = weight of the prepreg specimen, g and
W_4 = weight of the specimen after flow test, g.

9.2 Alternatively the resin flow may be calculated as a weight percentage of the volatile-free prepreg, namely:

$$RF \text{ (volatile}-\text{free)}, \% = \frac{W_1 - (W_2 - W_3) - W_4}{W_1 - (W_2 - W_3)} \times 100 \quad (2)$$

where:
W_2 = original weight of specimen assembly, g and
W_3 = weight of specimen assembly after heating, g.

10. Report

10.1 The report shall include the following:

10.1.1 Complete identification of the material, including the fiber type, fiber manufacturer, fiber treatment, resin identification, resin manufacturer, and manufacturer of the prepreg material,

10.1.2 Ply orientation and stacking sequence,

10.1.3 The resin flow in weight percent for each specimen and the average resin flow,

10.1.4 The actual test temperature,

10.1.5 The cure time used, and,

10.1.6 Deviations to this method if any.

11. Precision and Bias

11.1 *Precision*—The precision, defined as the degree of mutual agreement between individual measurements, can be estimated from the results of a round robin conducted on samples of prepreg by four laboratories, each laboratory making three measurements on each sample. The coefficient of variation for the total of twelve measurements having a mean resin flow of 26.1 % was 11.2 %. No modern measure of precision is available.

11.2 *Bias*—No estimate of bias can be offered as no accepted reference level is available.

12. Keywords

12.1 carbon fiber-epoxy prepreg; resin flow