Designation: D4102/D4102M – 22

Standard Test Method for Thermal Oxidative Resistance of Carbon Fibers[1]

This standard is issued under the fixed designation D4102/D4102M; 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 U.S. Department of Defense.

1. Scope

1.1 This test method covers the apparatus and procedure for the determination of the weight loss of carbon fibers, exposed to ambient hot air, as a means of characterizing their oxidative resistance.

1.2 *Units*—The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system are not necessarily exact equivalents; therefore, to ensure conformance with the standard, each system shall be used independently of the other, and values from the two systems shall not be combined.

1.2.1 Within the text, the 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazard information, see Section 8.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C613/C613M Test Method for Constituent Content of Composite Prepreg by Soxhlet Extraction

D3878 Terminology for Composite Materials

3. Terminology

3.1 *Definitions*—Terminology in accordance with Terminology D3878 shall be used where applicable.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *carbon fibers, n*—fibers produced by the pyrolysis of organic precursor fibers such as rayon, polyacrylonitrile (PAN), and pitch in an inert atmosphere; carbon fibers typically are carbonized at about 1300 °C [2400 °F] and assay at 93 % or more carbon.

3.2.2 *finish, n*—a material, with which filaments are treated, which contains a coupling agent to improve the bond between the filament surface and the resin matrix in a composite material; in addition, finishes often contain ingredients which provide lubricity to the filament surface, preventing abrasive damage during handling, and a binder which promotes strand integrity and facilitates packing of the filaments.

3.3 *Symbols:*

%CV—coefficient of variation
N—number of test specimens
s—standard deviation
W_a—specimen weight after the hot air exposure
W_d—specimen weight after drying
W_{dr}—percent weight loss in drying
W_e—final specimen weight after the finish removal
W_f—percent finish on the fiber
W_h—percent weight loss in hot air
W_i—specimen weight before finish removal
X—average
X_i—weight loss of the ith specimen

4. Summary of Test Method

4.1 The test method is composed of two parts. The first one specifies exposure conditions for an accelerated measurement, determining weight loss of the carbon fiber after 24 h in air at 375 °C [707 °F]. The second part specifies conditions for an extended measurement, determining the weight loss resulting from 500 h exposure in air at 315 °C [600 °F].

5. Significance and Use

5.1 The test is used to determine the oxidative resistances of carbon fibers as a means of selecting the most stable fibers for

[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, 2022. Published May 2022. Originally approved in 1982. Last previous edition approved in 2015 as D4102 – 82(2015). DOI: 10.1520/D4102_D4102M-22.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

incorporation in high-temperature fiber-reinforced composite systems. It can be used for quality control, material specification, and for research and development of improved carbon fibers. Factors that influence the oxidative resistance and should be reported are fiber identification, carbon fiber precursor type, fiber modulus, and any information on impurities, particularly metals. Also note that the presence of finish on the fiber can affect the oxidative resistance, and thus, alternative specimen preparations that enable the evaluation of finish effects are included.

6. Apparatus

6.1 The accuracy of all measuring equipment shall have certified calibrations that are current at the time of use of the equipment.

6.2 *Balance,* capable of weighing to the nearest 0.1 mg.

6.3 *Vacuum Oven,* capable of providing vacuum of 1.3 kPa [0.19 psi] or less at 80 ± 10 °C [177 ± 18 °F].

6.4 *Circulating Air Oven,* with sufficient flow rate and capability to change the ambient air in the chamber once a minute, while maintaining the required temperature within ±5 °C [±9 °F] in an operating range of 25 °C [77 °F] to 375 °C [707 °F].

6.5 *Glass Beakers,* borosilicate, 250 mL [8.45 oz], or other size, appropriate for the oven (one per sample).

6.6 *Wire Mesh Covers,* for the beakers to reduce excessive air turbulence during the exposure.[3]

6.7 *Boiling Flasks or Erlenmeyer Flasks,* borosilicate glass, 250 or 500 mL [8.45 to 16.91 oz] size, with standard-taper joint.

6.8 *Glass Condensers,* borosilicate for the above flasks.

6.9 *Hot Plate.*

6.10 *Tweezers,* stainless steel.

7. Reagents and Materials

7.1 *Methyl Ethyl Ketone* (2-butanone) 99.5 % pure, boiling range 70.0 to 81.0 °C [158 to 177.8 °F], or other suitable solvents recommended in Test Method C613/C613M.

8. Hazards

8.1 The methyl ethyl ketone, classified as an irritant and a fire hazard, should be handled in a well-ventilated area and should not be exposed to direct heat or open flame.

9. Test Specimen and Sampling

9.1 Using clean gloves to prevent any contamination, particularly with salt, unwrap the outer layers, which may have been contaminated by previous handling or environmental exposure, from the test package of carbon fiber yarn (or tow) and discard. Form a small coil of fresh fiber weighing approximately 2 g around two gloved fingers and tuck the ends in to obtain a specimen in the form of an easily handleable loop.

9.2 *Number of Specimens*—For quality control purposes, test a minimum of two specimens from each sample. For a quantitative assessment of the fiber performance, however, test a minimum of ten specimens and evaluate the results statistically as described in 12.4. It is recommended to report the method of sampling the specimens from the test package of carbon fiber yarn (or tow); for example, spacing of specimens or distribution of specimens along the length of the yarn (or tow), or both.

9.3 *Finish Removal From the Fiber*—Finish materials are generally present at about 1 % levels and are usually not stable at the exposure temperatures prescribed herein. The finish, if present, may be removed by extraction with hot solvent, such as methyl ethyl ketone or dimethyl formamide (DMF). Soxhlet extraction as described in Test Method C613/C613M is recommended for difficult-to-remove finishes and as a reference control. However, other finishes may be extracted by the procedure given in 11.3.1 – 11.3.4.

9.4 *Finish Left on the Fiber*—Since the fiber will normally be used with the finish intact, it is most useful to know the oxidative resistance of the fiber containing finish. To characterize and select fibers with optimum finishes, it is also very useful to know the relative effects of a variety of finishes. For this reason, it is desirable to have approaches for the determination of oxidative resistance both with and without finish.

10. Conditioning and Drying

10.1 Place the test specimens, beakers, and gauze covers in a vacuum oven at 77 ±10 °C [170 ±18 °F] and dry for 16 ± 1 h at a reduced pressure during the procedural steps described in 11.4.1 and 11.4.2.

11. Procedure

11.1 Weigh each specimen as removed from the sample package to the nearest 0.1 mg. Record the initial weight, W_i. In this and all subsequent weighings, use clean, dry stainless steel tweezers for the transfer of specimens.

11.2 If finish is to be removed (see 9.3), then either use a Soxhlet extraction as recommended in Test Method C613/C613M or follow Steps 11.3.1 – 11.3.4. If finish is not to be removed, skip Steps 11.3.1 – 11.3.4 and proceed with Step 11.4.

11.3 *Removal of the Finish:*

11.3.1 Put the specimen in a dry flask and cover with 100 to 200 mL [3.38 to 6.76 oz] of methyl ethyl ketone solvent. Place the condenser onto the flask and start the cooling water. Heat the flask on the hot plate or heating bath to bring the solvent to boil. Soak the specimen in the boiling solvent for 15 ± 1 min. Take off the condenser, decant the solvent, and remove the specimen.

11.3.2 Dry the specimen in the vacuum oven at 77 ± 10 °C [170 ± 18 °F] at a reduced pressure up to 1.3 kPa [0.19 psi] for 30 ± 1 min.

[3] 20 mesh nickel-chromium wire gauze from Fisher Scientific Co. has been found satisfactory for this purpose.

11.3.3 Weigh the dried specimen to the nearest 0.1 mg. Record the weight.

11.3.4 Repeat Steps 11.3.1 – 11.3.3 until the weight remains constant, within ±0.1 mg. Record the final weight, W_e.

11.4 *Drying:*

11.4.1 Dry each specimen for 16 ± 1 h in a vacuum oven at 77 ± 10 °C [170 ± 18 °F] at a reduced pressure up to 1.3 kPa [0.19 psi].

11.4.2 After drying, weigh the specimen to the nearest 0.1 mg and record the weight, W_d. Weigh each specimen in a tared beaker or crucible.

11.5 *Testing—Procedure A* (Short Term):

11.5.1 Preheat the air oven to 375 ± 5 °C [707 ± 9 °F] and make sure that the specimen positions in the oven are at 375 ± 5 °C [707 ± 9 °F] at the air circulation rate specified in 6.4.

11.5.2 Place the beakers with the specimens in the oven and record the starting time.

11.5.3 After 24.0 ± 1 h remove the specimens from the oven, cool in a desiccator, weigh to the nearest 0.1 mg, and record the weight, W_a.

11.6 *Testing—Procedure B* (Long-Term):

11.6.1 Preheat the air oven to 315 ± 5 °C [600 ± 9 °F] and make sure that the specimen positions in the oven are at 315 ± 5 °C [600 ± 9 °F] at the air circulation rate specified in 6.4.

11.6.2 Place the beakers with the specimens in the oven and record the starting time.

11.6.3 After 500.0 ± 1 h, remove the specimens and containers from the oven, cool in a dry atmosphere, weigh the specimens to the nearest 0.1 mg, and record the weight, W_a. (Weight losses can be obtained at intermediate times to obtain rate information.)

12. Calculations

12.1 *Fiber Finish*—Determine the amount of finish, in weight percent, as follows:

$$W_f = (W_i - W_e/W_i) \times 100 \quad (1)$$

where:

W_f = percent finish on the fiber,
W_i = specimen weight before finish removal, mg, and
W_e = final specimen weight after the finish removal, as in 11.3.4, mg.

12.2 *Weight Loss in Drying*—Calculate the weight loss in drying as follows:

$$W_{dr} = (W_e - W_d/W_e) \times 100, \text{ or} \quad (2)$$

$$(W_i - W_d/W_i) \times 100$$

where:

W_{dr} = percent weight loss in drying,
W_e = as in 12.1,
W_i = as in 12.1, and
W_d = specimen weight after drying, as in 11.4.2, mg.

12.3 *Weight Loss in Air Oxidation*—Calculate the relative oxidative weight loss, in weight percent, as follows:

$$W_h = (W_d - W_a/W_d) \times 100 \quad (3)$$

where:

W_h = percent weight loss in hot air,
W_a = specimen weight after the hot air exposure, mg, and
W_d = as in 12.2.

12.4 *Statistical Evaluation*—Calculate the average, X, the standard deviation, s, and the coefficient of variation, % CV, for each sample, comprised of ten or more tested specimens, as follows:

$$X = \frac{\sum_{i=1}^{n} X_i}{N} \quad (4)$$

$$s = \left[\frac{\sum_{i=1}^{N} (X_i - {}^{-}X)^2}{N-1} \right]^{1/2}$$

$$\% \text{ CV} = (s/X) \times 100$$

where:

N = number of test specimens, ≥10, and
X_i = weight loss of the ith specimen.

13. Report

13.1 The report shall include the following:

13.1.1 Complete identification of the material evaluated, including fiber type, source, manufacturer's code number(s) form, previous history, carbon fiber precursor type, type and nature of finish, and levels of impurities, if known.

13.1.2 Finish removal and conditioning procedures, if other than specified herein.

13.1.3 Number of specimens tested for given sample.

13.1.4 Identification of the test procedure used.

13.1.5 Weight loss in drying; average value, and standard deviation plus coefficient of variation if $N \geq 10$.

13.1.6 Percent finish on the fiber; average value, standard deviation, and coefficient of variation if $N \geq 10$.

13.1.7 Percent weight loss in air; average value, standard deviation, and coefficient of variation if $N \geq 10$.

13.1.8 Date of test.

14. Precision and Accuracy

14.1 No estimate of accuracy can be offered as no accepted reference level is available. The precision, defined as the degree of mutual agreement between individual measurements, cannot yet be estimated because of insufficient amount of data.

15. Keywords

15.1 carbon fiber; fiber finish; oxidation