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Standard Test Method for Fiber Content by Digestion of Reinforced Metal Matrix Composites¹

This standard is issued under the fixed designation D 3553; 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 digestion method for the determination of the fiber content of metal matrix composites. The test method used is based on the digestion of the matrix metal by a suitable acid or base that does not attack the fibers excessively (maximum 2 % weight loss).

1.2 *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 safety precautions, see Section 7.

2. Referenced Documents

2.1 ASTM Standards:

D 792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement²

3. Summary of Test Method

3.1 The test method consists of digesting the matrix metal of a weighed composite specimen. The digestion medium used depends upon the nature of the matrix and fiber. The residue is filtered, washed, dried, and weighed. The weight percent of fiber can then be converted to a volume percent if the fiber and composite densities are known. A correction for the weight loss of fiber, if any, may be made.

3.2 Three procedures are given: *Procedure A* for metals digestible by nitric acid; *Procedure B* for metals digestible by sodium hydroxide; and *Procedure C* for metals digestible by hydrochloric acid. Examples of metals coming under Procedure A are copper and steel, under Procedure B are aluminum, and under Procedure C are magnesium, titanium, steel, and copper.

4. Significance and Use

4.1 The fiber content of a composite must be determined to calculate the apparent strength and modulus of the reinforcing

fibers in the composite. Fiber content may be further used to evaluate the quality of a given composite specimen.

5. Apparatus

5.1 General:

5.1.1 *Sintered-Glass Filter*, fritted, medium porosity.

5.1.2 *Crucible Holder*.

5.1.3 *Analytical Balance*.

5.1.4 *Drying Oven*, capable of attaining 100°C.

5.1.5 *Glass Vacuum Filter Flask*.

5.1.6 *Vacuum Source*, capable of 13.3 kPa (100 mm Hg) pressure.

5.2 Procedure A:

5.2.1 *Flasks*, with ground-glass joints.

5.2.2 *Water Reflux Condenser*, with ground-glass joints.

5.2.3 *Water or Oil Bath*, temperature-regulated.

5.3 Procedures B and C:

5.3.1 *Calibrated Pipets*.

5.3.2 *Hot Plate*.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 Procedure A:

6.2.1 *Nitric Acid*, HNO₃ 65 % aqueous.

6.3 Procedure B:

6.3.1 *Sodium Hydroxide Solution*, NaOH 40 to 80 % aqueous.

6.4 Procedure C:

6.4.1 *Hydrochloric Acid*, HCl 5 to 10 % aqueous.

¹ This test method is under the jurisdiction of ASTM Committee D-30 on High Modulus Fibers and Their Composites and is the direct responsibility of Subcommittee D30.07 on Metal-Matrix Composites.

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² *Annual Book of ASTM Standards*, Vol 08.01.

³ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

7. Safety Precautions

7.1 Use standard procedures for handling hot acid.⁴

8. Test Specimens

8.1 Preferably five or more specimens, each weighing at least 300 mg, are cut from the sample of interest. The specimen may be of any convenient shape.

9. Procedures

9.1 *Procedure A*—Steel or copper matrices (and others that can be digested by HNO₃):

9.1.1 Weigh each specimen on an analytical balance to four significant figures.

9.1.2 If the fiber content by volume is required, determine the density of each specimen by a liquid displacement technique, in accordance with Test Methods D 792.

9.1.3 Place each specimen in a separate flask containing 30 mL of 65 % HNO₃.

9.1.4 Fit the flasks with reflux condensers and place them in a constant-temperature water or oil bath held at a temperature appropriate to the composite system.

9.1.5 When the digestion is complete, record the total digestion time for the purposes of 10.1. Filter the contents of each flask onto a tared sintered-glass filter under a vacuum of 13.3 kPa (100 mm Hg), or better. Wash the fiber with excess water followed by acetone.

9.1.6 Place the filter and specimen in an oven at 100 ± 10°C for 1 h to remove residual water and acetone.

9.1.7 Cool the filter containing the fiber residue to room temperature in a desiccator and accurately weigh the sample to four significant figures on an analytical balance.

9.2 *Procedure B*—Aluminum or brass matrices (and others that can be digested by NaOH solution):

9.2.1 Weigh each specimen on an analytical balance to four significant figures.

9.2.2 Determine the density of each specimen by a liquid displacement technique, as described in Test Methods D 792, if the fiber content by volume is required.

9.2.3 Place each specimen into a suitable beaker.

9.2.4 Add 20 to 25 mL of 40 to 80 % NaOH solution to the beaker. Place the beaker with the solution-specimen mixture on a hot plate and heat to 80°C.

9.2.5 Maintain at a temperature appropriate to the composite system until all the matrix metal is digested.

9.2.6 Record the total digestion time for the purposes of 10.2. Remove the beaker from the hot plate and allow the solution to cool.

9.2.7 Filter the contents of each beaker onto a tared sintered-glass filter under a vacuum of 13.3 kPa (100 mm Hg), or better. Wash the fibers with excess water followed by acetone.

9.2.8 Place the filter and specimen in an oven at 100 ± 10°C for 1 h to remove residual water and acetone.

9.2.9 Cool the filter containing the fiber residue to room temperature in a desiccator and accurately weigh the sample to four significant figures on an analytical balance.

9.3 *Procedure C*—Steel, titanium, copper, aluminum matrices (and others that can be digested by HCl):

9.3.1 Weigh each specimen on an analytical balance to four significant figures.

9.3.2 Determine the density of each specimen by a liquid displacement technique as described in Test Methods D 792, if the fiber content by volume is required.

9.3.3 Place each specimen into a suitable beaker.

9.3.4 Add 25 to 30 mL of 5 to 10 % HCl to the specimen. Place the beaker with the acid-specimen mixture on a hot plate and heat to a temperature appropriate to the composite system.

9.3.5 Continue to heat until all the matrix metal is digested.

9.3.6 Record the total digestion time for the purposes of 10.2. Remove the beaker from the hot plate and allow the solution to cool.

9.3.7 Filter the contents of each beaker onto a tared sintered-glass filter under a vacuum of 13.3 kPa (100 mm Hg), or better. Wash the fibers with excess water followed by acetone.

9.3.8 Place the filter and specimens in an oven at 100 ± 10°C for 1 h to remove residual water and acetone.

9.3.9 Cool the filter containing the fiber residue to room temperature in a desiccator and accurately weigh the sample to four significant figures on an analytical balance.

10. Correction for Fiber Weight Loss by Solvent

10.1 Weigh a bundle of fibers equal in size and number to the estimated amount present in the samples being digested on an analytical balance to four significant figures.

10.2 Repeat Procedure A (9.1), Procedure B (9.2) or Procedure C (9.3), whichever is applicable, with the above bundle of fibers digested for an equal length of time as the specimen.

10.3 The fiber weight loss by solvent action equals the combined weight of the filter and the starting fiber minus the combined weight of the filter and the remaining fiber. Only differences larger than 0.5 % should be considered significant.

NOTE 1—Certain fibers may, when in equilibrium with the humidity of the atmosphere, contain absorbed water up to a few percent by weight. Thus, conditioning and weighing in a controlled humidity environment should be employed to differentiate between fiber attack by the solvent and moisture pickup from the atmosphere.

11. Calculation

11.1 The weight of fiber in composite equals the combined weight of the filter and the residue as determined in 9.1.7, 9.2.9 or 9.3.9, plus the fiber weight loss to the solvent determined in 10.3 minus the tare of the filter.

11.2 Calculate the percent fiber in composite by weight:

$$\text{Weight \%} = w_F/w_c \times 100 \quad (1)$$

where:

w_F = weight of fiber in composite and

w_c = weight of initial composite specimen.

11.3 Calculate the percent fiber in composite by volume:

$$\text{Volume \%} = \frac{w_F/\rho_F}{w_c/\rho_c} \times 100 \quad (2)$$

where:

ρ_F = fiber density and

ρ_c = composite density

⁴ For example, see "Fire Protection Guide on Hazardous Materials," Fifth edition, 1973, available from the National Fire Protection Association, 470 Atlantic Ave., Boston, MA 02210.