



Standard Test Method for Determining the Particle Size Distribution of Advanced Ceramics by Centrifugal Photosedimentation¹

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1. Scope

1.1 This test method covers determination of the particle size distribution of advanced ceramic powders specifically silicon nitride and carbides, in the range of 0.1 to 20 μm, having a median particle diameter from 0.5 to 5.0 μm.

1.2 The procedure described in this test method may be applied successfully to other ceramic powders in this general size range, provided that appropriate dispersion procedures are developed. It is the responsibility of the user to determine the applicability of this test method to other materials.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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. Referenced Documents

2.1 *ASTM Standards:*²

C 242 Terminology of Ceramic Whitewares and Related Products

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions*—Refer to Terminology C 242 for definitions of terms used in this test method.

¹ This test method is under the jurisdiction of ASTM Committee C28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.03 on Physical Properties and Performance.

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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.

4. Summary of Test Method

4.1 A homogeneous aqueous dispersion of the powder is prepared. While kept in a thoroughly mixed condition, a small aliquot is transferred to the analyzer sample cell, which is placed in the instrument and subjected to a controlled centrifugal acceleration at a known or controlled temperature. At predetermined times related to the sedimentation of specific Stokes' diameters (Note 1), the optical absorbance is recorded and compared to the initial value to determine the fraction of the total sample that has sedimented a specific distance. A volume-based size distribution is calculated from the absorbance-time data. The results are reported as equivalent diameter (spherical) (Note 2) since particles in these powders are not truly spherical.

NOTE 1—This diameter in μm is referred to as D in the equation given below.

NOTE 2—Refer to Terminology C 242 for the ASTM definition of this term. Most equipment manufacturers refer to this as the equivalent spherical diameter.

$$D^2 = \frac{18\eta \cdot \log \frac{X_1}{X_2}}{(p_s - p_f)u^2 t} \times 10^8 \quad (1)$$

where:

η = viscosity of the fluid, cP

X_1 = distance from the middle of the centrifugal disc cell to the point in the cell where sedimentation starts,

X_2 = distance from the center of the centrifugal disc cell to the point where the light beam intersects the particle

t = time for the particle to settle, s

ρ_s = particle density, g/cm³,

ρ_f = fluid density, g/cm³, and

u = rotational angular velocity, rad/s.

4.2 The instruments that have been found suitable for this test method incorporate microcomputers that control instrument operation and perform all required data acquisition and computation functions.

5. Significance and Use

5.1 Manufacturers and users of advanced ceramic powders will find this test method useful for determining the particle size distribution of these materials for product specification, quality control, and research and development.

6. Apparatus

6.1 *Centrifugal Particle Size Distribution Analyzer*³—The analyzer shall incorporate a centrifuge capable of subjecting an homogeneous dispersion of the sample to centrifugal acceleration in specially designed sample cells. A collimated beam of visible light (either monochromatic or broad-band) shall traverse the sample cell at a defined distance from the top of the cell. The change in photo-extinction resulting from sedimentation of the sample shall be measured by a photo-detector and appropriate electronic circuits and used to calculate the volume-based size distribution of the sample.

6.2 *Ultrasonic Probe*, consisting of a 200 to 300-W power unit, ultrasonic transducer, and 13-mm (1/2-in.) diameter probe.

6.3 *Balance*, top-loading, accurate to 10 ± 0.1 g.

6.4 *Stirrer*, magnetic, with 25-mm (1-in.) and 19-mm (3/4 in.) stirring bars.

6.5 *Thermometer*, mercury or alcohol, 0 to 50°C, accurate to 0.5°C.

6.6 *Sample Cells*, as supplied by the instrument manufacturer.

7. Reagents

7.1 *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.⁴

7.2 *Darvan C⁵ Dispersing Fluid*—Dissolve 5.0 g Darvan C (ammonium salt of polymethacrylic acid) in 95 cm³ distilled water (Note 3).

NOTE 3—These reagents are adequate to cover the range of samples up to a maximum particle diameter of 20 μm.

7.3 *pH Adjusters*—Fresh ammonium hydroxide (NH₄OH) at 5 to 10 % strength is a common reagent used to raise pH while fresh nitric acid (HNO₃) at 5 to 10 % strength is a common reagent to lower pH.

8. Procedure

8.1 Sample Preparation and Dispersion:

8.1.1 Add the appropriate sample mass of powder, usually 1 g, to 250 cm³ dispersing medium in a 500-cm³ beaker. The dispersing medium consists of 250 cm³ of distilled water to which 0.3 g of dispersing liquid is added.

8.1.1.1 *Sample Dispersion*—Place the beaker containing sample in an ice water bath so that the temperature of the sample does not exceed approximately 30°C. Adjust the pH of the sample to 9.0 ± 0.2 to ensure adequate dispersion aided by the chemical environment. Deagglomerate the sample by inserting the ultrasonic transducer into the dispersing liquid/sample in a beaker. Ensure that the transducer is approximately 10.0 mm above the bottom of the sample beaker. Apply the ultrasonic energy at 40 W or higher for 1.0 min, and allow the sample to settle for 1.0 min. Repeat this procedure three times so that the total ultrasonic energy application time is 3 min. This high level of mechanical energy application is necessary to accomplish deagglomeration of the particles. The extent of deagglomeration is expected to be different for powders produced by different processes. An alternate approach is to ultrasonicate for 3 min continuously before pH and temperature check. Readjust the pH, if necessary, after ultrasonic application. Keep stirring the sample continuously, and test it as soon as practical to prevent reagglomeration.

8.1.2 Add a 25-mm (1-in.) stirring bar to the beaker, and place it on a magnetic stirrer. Stir for approximately 3 min in a cold water bath to bring the sample to ambient temperature. Continue stirring at constant temperature.

8.2 Analyzer Preparation:

8.2.1 To warm up the analyzer, apply power for a minimum of 10 min prior to testing. Conduct the warmup with the sample compartment closed. Make certain that the ventilation airflow is not restricted by adjacent equipment, papers, or other materials. Check the printer to ensure a sufficient supply of paper exists. Clean a pair of sample cells and caps, rinse with the 0.1 % Darvan C solution, and store inverted on absorbent paper.

NOTE 4—The concentration of the sample may require dilution with 0.1 % Darvan C solution to meet the optical absorbance tolerance specified in the instrument operating manual. Thorough mixing must accompany any dilution of the sample.

8.2.2 If required by the manufacturer's operating manual, check and adjust the zero and full-scale settings. These adjustments can be crucial to reliable and reproducible measurements.

8.2.3 Input the test parameters (sample and fluid density, fluid viscosity, test range, sedimentation distance, and centrifuge speed) according to the manufacturer's operating manual. Set the fluid viscosity at the value corresponding to the actual room temperature (see Table 1). Set the range such that the volume percent greater than the maximum diameter is certainly zero and the volume percent finer than the minimum diameter is less than 10 % (cumulative percent oversize greater than 90 %). Preliminary test runs may be required to establish the range and the size of the divisions necessary to achieve the test

³ Instruments from Horiba Instruments, Inc., 17671 Armstrong Ave., Irvine, CA 92714, have been found satisfactory. Other instruments may also be found suitable.

⁴ *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 *Reagent Chemicals and Standards*, by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the *United States Pharmacopeia*.

⁵ Darvan C is a trademarked product of R.T. Vanderbilt Company, Inc., Norwalk, CT 06856-5150.

TABLE 1 Viscosity Coefficients of Water^A

Temperature, °C	20	21	22	23	24	25	26	27	28	29
Viscosity coefficient, mPa*s or cP	1.01	0.98	0.96	0.94	0.92	0.89	0.87	0.86	0.84	0.82

^AViscosity coefficient values to two decimal places have been found satisfactory for this test method.

limits. The test range and division settings shall provide a minimum of ten divisions within the test range.

8.2.4 If required by the manufacturer's operating manual, conduct a blank test with clean dispersant fluid in the sample and reference cells.

8.3 Test Performance:

8.3.1 Adjust the rate of the magnetic stirrer to produce a slight vortex in the sample dispersion. Withdraw an appropriate volume of sample from the center of the dilute dispersion with a disposable plastic pipet. Make certain that the dispersion is mixed thoroughly by vigorous pumping with the pipet (avoid strong agitation, which would create bubbles). Completely transfer the withdrawn sample to the drained sample cell. Insert the cell cap, carefully wipe all outer surfaces of the cell, place in the centrifuge, and start the test. Monitor the absorbance display on the instrument to ensure that the initial absorbance is within the specified tolerance at the beginning of the test. If not, refer to Note 4. When the test is completed, remove the sample cell, clean immediately, and rinse with dispersant fluid.

9. Presentation of Data

9.1 *Analyzer Printout*—The analyzers that have been found suitable for this test method render data printouts that provide a permanent record of the test conditions and graphic records of the size distribution. These printouts may provide sufficient information for many purposes.

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10. Fluid Densities and Viscosity Coefficients

10.1 Table 1 lists the viscosity coefficients for water over the temperature range of 20 to 29°C. Use these values for the Darvan C solution. Use a density of 1.00 g/cm³ for these solutions.

11. Additional Data Presentations

11.1 The analyzers that have been found suitable for use with this test method produce a printed report that may include a cumulative size distribution table showing the volume percent oversize, starting with the programmed maximum diameter. The incremental divisions are selectable by the operator in some operating modes and, in others, determined by the microcomputer in the instrument.

12. Precision and Bias

12.1 *Precision*—No statement of precision can be made since round-robin testing has not been completed.

12.2 *Bias*—It is not possible to discuss the bias of the results obtained using this test method since no absolute method of particle size distribution is recognized.

13. Keywords

13.1 advanced ceramics; particle size distribution; photosedimentation