Designation: C 1274 – 00

Standard Test Method for Advanced Ceramic Specific Surface Area by Physical Adsorption[1]

This standard is issued under the fixed designation C 1274; 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.

1. Scope

1.1 This test method covers determination of surface area of advanced ceramic materials. This test method specifies general procedures that are applicable to many commercial physical adsorption instruments. This test method provides specific sample outgassing procedures for listed materials, including silicon carbide, silicon nitride, and zirconium oxide. It includes additional general outgassing instructions for other advanced ceramic materials. The multipoint equation of Brunauer, Emmett and Teller[2] (BET) along with the single point approximation of the BET equation form the basis for all calculations.

1.2 This test method does not include all existing procedures appropriate for outgassing advanced ceramic materials. The included procedures provided acceptable results for samples analyzed during round robin testing. The investigator must determine the appropriateness of listed procedures.

1.3 This test method uses SI units as standard. State all numerical values in terms of SI units unless specific instrumentation software reports surface area using alternate units. In this case, present both reported and equivalent SI units in the final written report. Many instruments report surface area as m^2/g, instead of using correct SI units (m^2/kg).

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:* [3]

D 1993 Test Method for Precipitated Silica—Surface Area by Multipoint BET Nitrogen Adsorption

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

3. Terminology

3.1 *Definitions:*[4]

3.1.1 *adsorbate,*, *n*—material that has been retained by the process of adsorption.

3.1.2 *adsorbent,*, *n*—any solid having the ability to concentrate significant quantities of other substances on its surface.

3.1.3 *adsorption,*, *n*—a process in which fluid molecules are concentrated on a surface by chemical or physical forces, or both.

3.1.4 *adsorptive,*, *n*—any substance available for adsorption.

3.1.5 *aliquant,*, *n*—a representative portion of a whole that divides the whole leaving a remainder.

3.1.6 *outgassing,*, *n*—the evolution of gas from a material in a vacuum or inert gas flow, at or above ambient temperature.

3.1.7 *physical adsorption (van der Waals adsorption),*, *n*—the binding of an adsorbate to the surface of a solid by forces whose energy levels approximate those of condensation.

3.1.8 *surface area,*, *n*—the total area of the surface of a powder or solid including both external and accessible internal surfaces (from voids, cracks, open porosity, and fissures). The area may be calculated by the BET (Brunauer, Emmett, and Teller[2]) equation from gas adsorption data obtained under specific conditions. It is useful to express this value as the specific surface area, for example, surface area per unit weight of sample (m^2/g).

3.1.9 *surface area (BET),*, *n*— the total surface area of a solid calculated by the BET (Brunauer, Emmett, Teller[2]) equation, from nitrogen adsorption or desorption data obtained under specific conditions.

3.1.10 *surface area, specific,*, *n*—the area, per unit mass of a granular or powdered or formed porous solid, of all external plus internal surfaces that are accessible to a penetrating gas or liquid.

4. Summary of Test Method

4.1 An appropriate sized sample (to provide at least the minimum surface area required for reliable results for the

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[2] Brunauer, S., Emmett, P. H., and Teller, E., *J. Am. Chem. Soc. 60*, 1938, pp. 309–319.

[3] 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] *Compilation of ASTM Standard Terminology*, 8th ed, 1994.

instrument or apparatus used) is outgassed under appropriate conditions prior to analysis.

4.2 *(Multipoint BET Analyses Only)*— Volume of gas adsorbed, or desorbed, is determined for a minimum of four relative pressures within the linear BET transformation range of the physical adsorption, or desorption, isotherm characteristic of the advanced ceramic. The linear range is that which results in a least square correlation coefficient of 0.999 (preferably 0.9999) or greater for the linear relationship used in the BET graph (1⁄(adsorbed volume * (1/relative pressure — 1))). Typically, the linear range includes relative pressures between 0.05 and 0.30, however, microporous materials usually require use of a range of lower relative pressures, such as 0.01 to 0.10.

4.3 *(Single Point BET Analyses Only)*— Volume of gas adsorbed, or desorbed, is determined at the highest known relative pressure within the linear BET transformation range of the physical adsorption, or desorption, isotherm. Typically, a relative pressure of 0.30 is used. (It may be necessary to perform a multipoint analysis of the material first to determine the optimum single point relative pressure.)

4.4 The sample is accurately weighed (to at least 1% of the sample mass) after analysis. It is important to use an analytical balance to determine the sample weight. The physical adsorption instrument or apparatus measures the total amount of gas adsorbed onto, of desorbed from, the sample under analysis. The sample weight is then used to normalize the measured results. Any error in the sample weight will be propagated into the final BET surface area.

4.5 Calculations are based on the BET equation, as required by the instrument being used for the determination. The cross sectional area for the adsorbate is taken to be 0.162 nm^2 if nitrogen is used as the adsorptive. Use the appropriate value recommended by the instrument manufacturer for adsorptives other than nitrogen. Report this cross sectional area with the BET surface area results.

5. Significance and Use

5.1 Both suppliers and users of advanced ceramics can benefit from knowledge of the surface area of these materials. Results of many intermediate and final processing steps are controlled by, or related to, specific surface area of the advanced ceramic.

6. Interferences

6.1 This test method can be used to determine the internal and external surface of a powder or solid only after these surfaces have been cleaned of any physically adsorbed molecules. Such adsorbed species, for example, water or volatile organic compounds, prevent physical adsorption of the gas probe molecules used to measure surface area. Therefore, it is necessary to remove these adsorbed contaminants prior to surface area analysis. Generally, such outgassing is performed by evacuating or flushing the sample. Outgassing can be accelerated by using elevated temperatures, provided no irreversible sample changes occur. Typical minimum vacuum levels attained are 10^{-1} Pa. Typical flushing gases are helium, nitrogen, or a mixture of the two. Outgassing is complete when duplicate surface area analyses produce results within expected instrument repeatability limits, when a constant residual vapor pressure is maintained upon isolation from the vacuum source, or when flushing gas composition is unaffected while passing over the sample.

7. Apparatus

7.1 *Classical Vacuum Apparatus*—Refer to Test Method D 1993 for apparatus description.

7.2 *Automated and Dynamic Flow Instruments*— Commercial instruments are available from several manufacturers for the measurement of specific surface area by physical adsorption. Some are automated versions of the classical vacuum apparatus. Others may use a gravimetric technique to determine the amount of adsorbed gas on the sample surface. Additionally, commercial instruments are available which measure physical adsorption based on the dynamic flow method.

8. Reagents and Materials

8.1 *Liquid Nitrogen*.

8.2 *Nitrogen*, 99.99 mole percent, with the sum of O_2, Ar, CO_2, hydrocarbons (as CH_4), and H_2O totaling less than 10 ppm, dry and oil-free, cylinder, or other source of purified nitrogen.

8.3 *Helium*, 99.99 mole percent, with the sum of N_2, O_2, Ar, CO_2, hydrocarbons (as CH_4), and H_2O totaling less than 10 ppm, dry and oil-free, cylinder, or other source of purified helium, if needed for determination of void space above sample.

8.4 *Blended Nitrogen and Helium*, dry and oil-free, cylinder, or other source of blended gases. The actual composition of the blend must be known. For use with dynamic flow instruments only.

9. Sampling, Test Specimens, and Test Units

9.1 No specific instructions are given. However, it is important that the aliquant being analyzed represent the larger bulk sample from which it is taken. The bulk sample should be homogenized before any sampling takes place. Best results are obtained when a flowing bulk material is temporarily diverted into a collector for an appropriate time. It is better to sample the entire flow for a short time than to sample a portion of the flow for a longer time. Collecting several small aliquants and combining them improves the reliability of the sampling process. Rotating rifflers are available that satisfy these requirements.

10. Calibration and Standardization

10.1 *Classical Vacuum Apparatus*—Refer to Test Method D 1993 for calibration procedures.

10.2 *Automated and Dynamic Flow Instruments*—Follow manufacturer's instructions for calibration and operational verification of the instrument.

11. Outgassing

11.1 *Classical Vacuum Apparatus*—Refer to Test Method D 1993 for outgassing procedures.

11.2 *Automated and Dynamic Flow Instruments*:

11.2.1 Weigh (to the nearest 0.1 mg) clean, empty sample tube, along with stopper or seal. Record the empty tube weight.