Designation: D 3849 – 95a (Reapproved 2000)

Standard Test Method for Carbon Black — Primary Aggregate Dimensions from Electron Microscope Image Analysis[1]

This standard is issued under the fixed designation D 3849; 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 the morphological characterization of carbon black primary aggregates from transmission electron microscope images. The measurements are applicable to carbon blacks in the dry (as manufactured) state, extracted from unvulcanized rubber compounds and in a cellulose acetate butyrate paint chip dispersion.

1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information 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. Referenced Documents

2.1 *ASTM Standards:*

D 297 Test Methods for Rubber Products—Chemical Analysis[2]

D 1416 Test Methods for Rubber from Synthetic Sources—Chemical Analysis[2]

D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets[2]

D 3191 Test Methods for Carbon Black in SBR (Styrene-Butadiene Rubber)—Recipe and Evaluation Procedures[2]

D 3192 Test Methods for Carbon Black Evaluation in NR (Natural Rubber)[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *Aggregate Dimensional Properties from Image Analysis:*

3.1.1.1 *area (A)*—the two-dimensional projected area of the carbon black aggregate image.

3.1.1.2 *chord*—the length of a scanning intercept across an aggregate in a given direction. The *mean chord* (π *A/P*) is the average width of an aggregate in all directions.

3.1.1.3 *Feret's diameter*—the maximum spacing between parallel tangents to an aggregate in a given direction. The average Feret (*L*) is derived from an average of multiple measurements at specific angular increments.

3.1.1.4 *length (L_1)*—the longest Feret's diameter of an aggregate.

3.1.1.5 *perimeter (P)*—the total boundary length of an aggregate.

3.1.1.6 *projected length (L_2)*—the total length of an aggregate in a given direction, including the contribution of multiple entrants. The projected length is equal to the number of scan lines multiplied by a calibration factor that is equal to the scan line spacing in the proper dimensional units (usually nanometres for carbon blacks).

3.1.1.7 *volume*—the volume of carbon black aggregates may be measured directly by well-calibrated scanning microdensitometry (*V*) or geometrically (V_1) as follows:

$$V_1 = 8A^2/3P \quad (1)$$

3.1.1.8 *width*—the width of a carbon black aggregate may be described in terms of either the mean chord or Feret's diameter. The average width (*W*) is defined as the mean chord measured in a direction that is perpendicular to the longest projection. *W* is equal to the projected area divided by the longest projection. The average nondirectional width (W_1) is equal to the mean chord (π *A/P*). The shortest width (W_2) is equal to the shortest chord length and is derived from chord sizing. The longest width (W_3) is the shortest Feret's diameter from multiple measurements in different directions.

3.1.2 *Aggregate Nondimensional Shape Parameters*

3.1.2.1 *circularity factor (C.F.)*—the amount of deviation of the two-dimensional projected aggregate area from a circle expressed as follows:

$$C.F. = P^2/4\pi A \quad (2)$$

3.1.2.2 *form factor*—the length/width ratio of the aggregate. Some of the more commonly used ratios are as follows:

$$F_1 = L_1/W_1 \quad (3)$$

$$F_2 = L/W_1 \quad (4)$$

[1] This test method is under the jurisdiction of ASTM Committee D24 on Carbon Black and is the direct responsibility of Subcommittee D24.81 on Carbon Black Microscopy.

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[2] *Annual Book of ASTM Standards*, Vol 09.01.

$$F_3 = L_1/W_3 \quad (5)$$

3.1.2.3 *sphericity factor (S.F.)*—The amount of deviation of the projected aggregate image from a sphere expressed as follows:

$$S.F. = P^3/6\pi^2V \quad (6)$$

3.1.3 *carbon black aggregate*—a discrete, rigid colloidal entity that is the smallest dispersible unit; it is composed of extensively coalesced particles.

3.1.4 *carbon black particle*—a small spheroidally shaped, paracrystalline, non-discrete component of an aggregate; it is separable from the aggregate only by fracturing.

3.1.5 *chord sizing*—an operation in which a specified length increment (ΔL) is cut off each detected chord in an image. All chords shorter than ΔL are completely eliminated from the image and all chords larger than ΔL are shortened by ΔL. The operation is repeated over a range that eventually eliminates all of the chords in the image and thereby provides a chord size distribution.

3.1.6 *detected image*—an electronic monitor display of the chords across the features in a given field. The detected image should match the actual image as closely as possible.

3.1.7 *epidiascope*—a device for projecting images of photographic prints, negatives, or transparencies on the scanner tube.

3.1.8 *feature*—areas within a single continuous boundary (for example, an aggregate image) that have an optical-density value (gray-level range) that is distinct from the background area outside the feature.

3.1.8.1 *feature cropping*—the splitting of features at the boundaries of the measuring frame that, if uncorrected, results in erroneously small size values for these features.

3.1.9 *fiber optics coupling*—bundles of small-diameter light-channeling fibers that transmit the optical image from the fluorescent viewing screen within an electron microscope to the scanner of the image analysis system with a minimal loss of brightness.

3.1.10 *glow discharge*—a plasma of ionized gas that is formed in a high-voltage field at pressures of about 3 to 20 Pa (25 to 150 $\times 10^{-3}$ torr). An alternating current (a-c) glow discharge using air is effective in cleaning and oxidizing the surface of carbon substrates to improve the wetting characteristics of polar vehicles containing pigment dispersions.

3.1.11 *gray level*—variations in the intensity of images in terms of the electrical output of the scanner. The brightest region in an image gives the highest electrical output and is defined as" white," while the complete absence of light in a field is" black." The tone of detected features usually ranges between these extremes and the electrical output signal is known as the gray level.

3.1.11.1 *gray-level discrimination*—the ability to distinguish between different gray levels within features or between different features in a field. The gray levels within carbon black images in the electron microscope become lower with diminishing aggregate size.

3.1.12 *image analysis*—measurement of the size, shape, and distributional parameters of feature images by electronic scanning methods.

3.1.12.1 *feature specific*—image analysis data output that provides individual measurements on each separate feature. A multiparameter feature specific system enables the linking of different type measurements for each separate feature, thus enabling direct calculation of multivariate functions such as F, P^2/A, etc.

3.1.12.2 *field specific*—an image analysis data output that provides only field totals for each measured parameter. Number average measurements are obtained by dividing the total measured parameter by the feature count.

3.1.12.3 *off-line*—this type of image analysis system is based on scanning of negatives, transparencies, or photographic prints of the features utilizing an epidiascope or similar optical device.

3.1.12.4 *on-line*—a type of image analysis system in which the scanner is a part of the microscope or directly coupled to the microscope.

3.1.13 *microdensitometer*—an image analysis device for resolving gray-level differences within or between features and for integrating the optical density across scanned images of irregularly shaped objects. The latter provides three-dimensional size measurements (volume) of the particles or aggregates of noncrystalline materials such as silicas, or poorly crystallized materials such as carbon black.

3.1.14 *shading*—variation in the electrical output from the scanner from areas of identical gray level in different parts of the image. Shading can be due to optical effects, scanner deficiencies, or to artefacts in the specimen. A shading compensator is employed to correct any instrumental deficiencies.

3.1.15 *specimen anticontamination device*—a cold trap (cooled by liquid nitrogen) that is located in the vicinity of the specimen in an electron microscope in order to prevent the deposition of contaminants, such as diffusion pump oil vapor from the vacuum system, on the specimen.

3.1.16 *specimen grid*—a specimen mount in the form of a thin circular mesh about 3 mm in diameter that fits the standard specimen holders of transmission electron microscopes. Grids are used to support the thin substrates required for electron microscopy and are made most commonly of copper. Tungsten grids are used when the specimen must be heated at elevated temperatures.

3.1.17 *substrate*—a thin cast or vacuum-evaporated film that is used to support electron microscope specimens. Evaporated carbon films are a commonly used substrate because of relatively good mechanical strength, stability, and conductivity.

4. Significance and Use

4.1 Carbon black primary aggregate morphology significantly affects the transient and end-use properties of black loaded polymer systems. Vulcanizate hysteresis and strength properties (tear, tensile, and abrasion resistance) increase with diminishing aggregate size. Extrusion die swell diminishes and vulcanizate modulus increases with increasing aggregate irregularity (for example, the amount of deviation from a spherical shape).

4.2 Carbon black aggregate dimensional and shape properties are dependent upon the nature of the system in which the sample is dispersed, as well as the mixing procedure.

5. Apparatus

5.1 *Electron Microscope*, transmission-type, with a point-to-point resolving power of 1.0 nm or better. Operating voltages (electron beam) should include settings of 40 and 60 kV. The specimen chamber should contain an anticontamination device.

5.2 *Image Analysis System*, television scanner-type. The system shall also include a television monitor for viewing the specimens, a well-defined measuring frame that is visible on the monitor and has the capability of correcting for the effects of feature cropping at the borders, a compensator for eliminating shading effects, a detector for discerning the boundaries of the carbon black aggregates at different gray levels, and one or more computer modules for converting the output of the detector into dimensional information. Minimum requirements for dimensional output are area, perimeter, average Feret's diameter, and feature count. Desirable additional outputs are volume by microdensitometry and Feret's diameters in different directions. The system shall contain one or more devices for automated data recording, processing, averaging, and printing of results. Acceptable recorder-processing systems include an on-line computer, desk-top calculator, or a magnetic tape recorder-computer combination. The image analysis system may be field or feature specific, the latter being preferred. The system may be off-line or on-line. For the latter, a fiber optics coupling between the scanner and electron microscope is recommended.

5.3 *Vacuum Evaporator*, [3] standard-type, for preparing carbon films to be used as substrates for electron microscopy. The evaporator should be capable of reducing the absolute pressure to 1.3 mPa (1×10^{-5} torr) and should also contain the necessary apparatus for a-c glow discharge.

5.4 *Ultrasonic Generator*:

5.4.1 *Dispersion Procedures A and C*—Variable power tank-type ultrasonic cleaning unit,[4] 80 kHz, 100 W.

5.4.2 *Dispersion Procedure B*—Probe-type ultrasonic generator,[5] 20 kHz, 150 W.

5.5 *Dry Box*, capable of maintaining a relative humidity level of no greater than 30 %.

5.6 *Analytical Balance*, with an accuracy of about 0.5 mg.

5.7 *Combustion Furnace and Tube*—meeting the requirements described in Methods D 1416 or D 297.

5.8 *Carbon Rods*, [6] approximately 3.1 mm in diameter.

5.9 *Carbon Rod Sharpener*. [7]

5.10 *Glass Microscope Slides*, 25 by 75-mm.

5.11 *Test Tubes*, 75 by 10-mm, 4-cm^3 capacity, 0.5-mm wall thickness, with corks.

5.12 *Transfer Pipets*, disposable Pasteur-type, 225 mm long, 1-mm inside diameter at tip.

5.13 *Rubber Bulbs*, for pipets.

5.14 *Glass Vials*, 40-cm^3 capacity, with solvent-resistant tops.

5.15 *Glass Tubes*, straight wall, flat bottom, 90 mm in height, 26 to 27-mm inside diameter.

5.16 *Glass Jars*, 30-cm^3 capacity, wide-mouth with solvent-resistant caps, height and outside diameter approximately 43 mm.

5.17 *Glass Dishes*, two 185 mm in diameter, 100 mm in height.

5.18 *Büchner Funnel*, No. 3, 111-mm inside diameter.

5.19 *Vinyl Tubing*, approximately 50 mm long, 12.5-mm inside diameter.

5.20 *Clamp*, hose cock, open-jaw type.

5.21 *Filter Paper*, 125-mm diameter, fast.

5.22 *Electron Microscope Specimen Grids*, 3-mm diameter, 300-mesh copper.

5.23 *Electron Microscope Specimen Grids*, 3-mm diameter, 200-mesh tungsten.

5.24 *Specimen Grid Holders*. [8]

5.25 *Test Tube Holders*, for 48 tubes up to 16 mm in outside diameter.

5.26 *Wire Screening*, with openings approximately 1 mm^2.

5.27 *Forceps*, fine-tipped, locking-type.

5.28 *Tweezers*, fine-tipped.

5.29 *Spatulas*, micro-type with V-shaped spoon that is approximately 2 mm wide at top and 12.5 mm long.

5.30 *Solvent Dispenser*, portable high-speed type.

5.31 *Fluorocarbon Duster*. [9]

5.32 *Lens Tissue*, [10] lint-free.

5.33 *Porcelain Boats*, for pyrolysis, 98 mm long, 15 mm wide at top.

5.34 *Centrifuge*, 2094 rad/s (20 000 r/min) with head for 75 by 10 mm test tubes.

5.35 *Test Tubes*, polypropylene, 75 by 10 mm, 5 cm^3 capacity, 0.5 mm wall thickness with caps.

5.36 *Beakers*, 2000 cm^3 capacity.

6. Reagents and Materials

6.1 *Castor Oil*, laboratory grade.

6.2 *Chloroform*, reagent grade.

6.3 *Collodion*, typical commercial grade, U.S.P.

6.4 *1,2-Dichloroethane*, reagent grade.

6.5 *Ethyl Acetate*, reagent grade.

6.6 *Poly (Vinyl Formal) Resin*,[11] Grade 15/95.

6.7 *Cellulose Acetate Butyrate Resin*. [12]

6.8 *Phthalate Type Plasticizer*. [13]

[3] The following vacuum evaporator systems have been found to be acceptable: Denton Model DV-515, available from Denton Vacuum, Inc., Cherry Hill Industrial Center, Cherry Hill, NJ 08034; Ladd Vacuum Evaporator, Catalog No. 40000, Ladd Research Industries, Inc., P.O. Box 901, Burlington, VT 05401.

[4] Ladd Research Industries, Inc., P.O. Box 901, Burlington, VT 05401, Catalog No. 12400, is satisfactory.

[5] Branson Instruments, Inc., Model No. 185E, is satisfactory.

[6] Denton Vacuum Inc., Cherry Hill Industrial Center, Cherry Hill, NJ 08034, Catalog No. 5095-002, is satisfactory.

[7] Ernest F. Fullam, Inc., P.O. Box 444, Schenectady, NY 12301, Catalog No. 1204, is satisfactory.

[8] L.K.B. Instruments Co., 12221 Parktown Dr., Rockville, MD 20852, Catalog No. 4828B, is satisfactory.

[9] Ernest F. Fullam, Inc., P.O. Box 444 Schenectady, NY 12301, Catalog No. 1180-1, is satisfactory.

[10] Ladd Research Industries, Inc., P. O. Box 901, Burlington, VT 05401, Catalog No. 12700, is satisfactory.

[11] Formvar, a registered trademark of Monsanto, also available as Catalog No. 18050 and 18060, Ladd Research Industries, Inc., P.O. Box 901, Burlington, VT 05401.

[12] Eastman CAB 381-2 is satisfactory.

[13] Monsanto Santicizer 160 is satisfactory.

7. Sampling

7.1 *Dry Carbon Blacks*—Select vial size samples at random from larger size lots. The samples may be in either pelletized or nonpelletized form. Cap and label the vials for storage.

7.2 *Carbon Black in SBR* (*Styrene-Butadiene Rubber*) *and NR* (*Natural Rubber*)—Prepare the rubber compounds in accordance with the formulations and mixing procedures described in Test Methods D 3191 or D 3192. Sheet out in accordance with Practice D 3182 but do not vulcanize. Label the slabs and store at or below room temperature. A freezer (0°C) is recommended for long-term storage of unvulcanized rubber compounds.

7.3 *Carbon Black in CAB* (*Cellulose Acetate Butyrate*):

7.3.1 Weigh 160 g of carbon black, 176 g of CAB, and 64 g of plasticizer and blend by stirring in a beaker until the mixture becomes a uniform, free flowing powder.

7.3.1.1 The 160 g loading range (40 % by weight) is applicable for the majority of rubber grade carbon blacks but is not a critical aspect of the procedure.

7.3.1.2 Lower loadings in the range of 80 g (25 % by weight) generally provide easier mixing for the finer N100 and N200 grades or conductive types such as N472.

7.3.2 Mix the carbon black into the CAB resin by milling on a two-roll mill (200 by 460 mm) with a nip setting of 0.28 mm.

7.3.3 Regulate the temperature of the front roll of the mill at 82°C and the rear roll at 62°C.

7.3.4 Fuse the CAB-carbon black powder together on the mill (<1 min) and mix for a total of 10 min.

7.3.5 Cool the mix to room temperature and remix for 5 min. Repeat this procedure and sheet out the mix into thin slabs. Label the slabs and store at room temperature.

8. Samples and Test Specimens

8.1 *Samples*:

8.1.1 *Dry Carbon Blacks—Dispersion Procedure A*—Weigh 8 to 10 mg of black, place in a test tube containing 1 cm^3 of chloroform (from dispenser), and cork. Repeat this procedure for two more different samples from the same vial of black.

NOTE 1—With experience, it is not necessary to weigh each black sample. One full scoop with the microspatula provides a satisfactory amount of black for all samples.

8.1.2 *Dry Carbon Blacks—Dispersion Procedure B*—Weigh 7 mg of black and place in a vial containing 30 cm^3 of collodion solution prepared by mixing 1 cm^3 of collodion, 45 cm^3 of ethyl acetate, and 0.05 cm^3 (1 drop) of castor oil. Repeat the sampling procedure for two more samples from the same vial of black.

8.1.2.1 There is considerable latitude in the amount of black used. The 7-mg figure is based on the average rubber reinforcing-type black. The finer N100 and N200 blacks require somewhat less black and the coarser semireinforcing types require considerably more. Coarse blacks in the N700 to N900 classes require about 40 to 50 mg per 30 cm^3 of vehicle. Also, high DBP (dibutyl phthalate) absorption blacks require somewhat more sample and low DBP absorption blacks somewhat less sample.

8.1.3 *Carbon Blacks in SBR and NR, Dispersion Procedure C*—Cut an elongated thin section (about 4 mm^2 in cross-sectional area) of the unvulcanized rubber compound using a razor blade. From this, weigh a 75-mg sample and cut into small fragments that are approximately 1 mm.[3] Repeat this procedure for two more samples from different parts of the same rubber compound.

NOTE 2—With experience, it is not necessary to weigh the rubber sample. A20-mm long sample with a cross-sectional area of approximately 4 mm^2 is adequate.

NOTE 3—This is the recommended procedure for a large batch of rubber compound containing the same carbon black. Ideally, each of the three samples should be selected from a different slab. For small test batches, however, one sample from a single slab is considered adequate.

8.1.4 *Carbon Black in CAB, Dispersion Procedure D*—Cut an elongated thin section from the CAB compound in accordance with 8.1.3. From this, weigh a 25 mg sample and place in a glass test tube containing 1 cm^3 of ethyl acetate and cork.

8.1.4.1 There is considerable latitude in the mass of the sample. With experience, this can be estimated satisfactorily.

8.2 *Test Specimens*:

8.2.1 *Substrate Preparation*—Prepare thin backing films by wiping a clean glass slide with lint-free lens tissue. Wipe three times with one sheet of lens tissue and then repeat with a fresh sheet. Dip the slide into a 0.25 % solution of poly (vinyl formal) in 1,2-dichloroethane (Fig. 1(*a*)). Drain the dipped slide vertically on lens tissue until the film dries (about 1 to 2 min). Then, score all the edges of the film by rubbing a razor blade around the top edges of the slide. Blow away all film fragments using the fluorocarbon duster.

8.2.1.1 Carefully float the poly (vinyl formal) film on to a distilled water surface in the Büchner funnel (Fig. 1(*b*)). The water is held in the funnel by means of a small piece of vinyl tubing and clamp at the bottom. Place the 300-mesh copper specimen grids (shiny side up) one at a time on the top of the floating poly (vinyl formal) film (Fig. 1(*c*)). Generally, a total of 70 or more grids are prepared in a single operation.

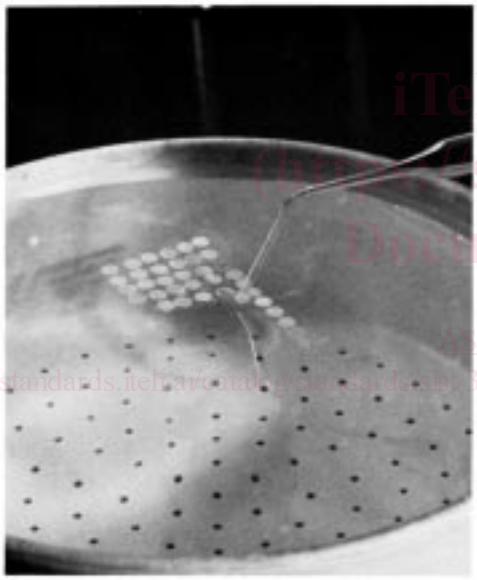
8.2.1.2 Prepare a small screen platform and place face down on the grids as shown in Fig. 1(*d*). Remove the grids and poly (vinyl formal) film from the water by depressing the screen platform and rotating it 180° through the water (Fig. 2(*a*)). An alternative procedure, used for the heavier tungsten grids, requires that the screen platform be placed under the water at the bottom of the Büchner funnel. The grids are deposited on the platform, shiny side down. Next, the poly (vinyl formal) film is positioned over the grids and allowed to settle on them by removing the water from the funnel. This procedure is applicable to either tungsten or copper grids, but the surface deposition and inversion method is simpler and considerably quicker. Allow the coated grids on the screen platform to dry at least 3 to 4 h before using them. Preferably, prepare the films in the afternoon and allow to dry overnight.

NOTE 4—In preparing poly (vinyl formal) films on 200-mesh tungsten grids, it is preferable to make them somewhat thicker so that they do not sag into the larger grid openings. A solution of 0.5 % poly (vinyl formal) in 1,2-dichloroethane is satisfactory for 200-mesh grids. Prior to use, the uncoated tungsten grids should be heated to 800°C, in accordance with Methods D 1416 or D 297, to remove any contaminants.

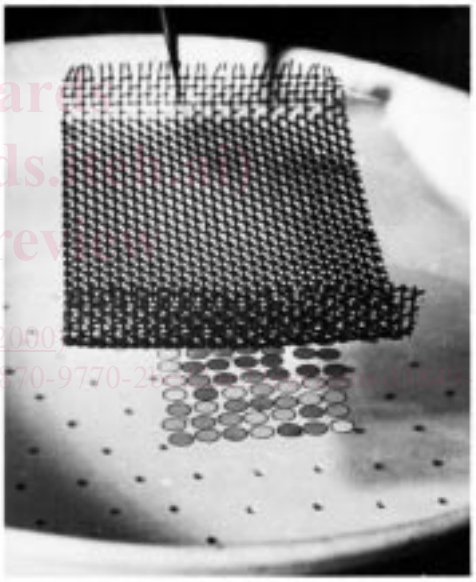
8.2.1.3 Place the screen platform containing the poly (vinyl formal) coated grids in a vacuum evaporator that has been set

(*a*) Dipping Slide in Poly (Vinyl Formal) Solution

(*b*) Floating Film onto a Water Surface



(*c*) Placing Specimen Grids on Film with Vacuum Pipet



(*d*) Placing Screen Platform on Floating Film with Grids

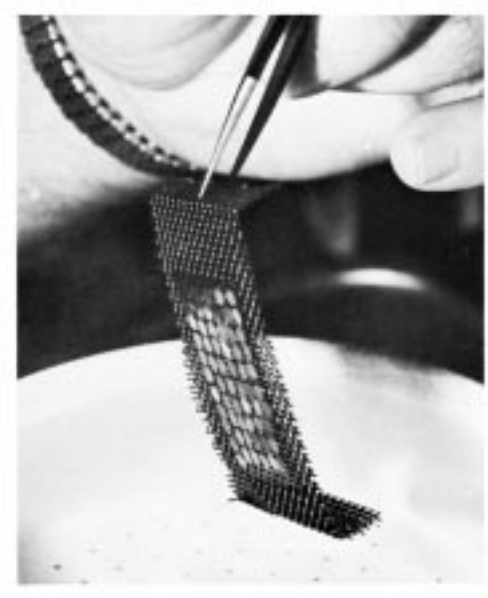
FIG. 1 The Preparation of Poly (Vinyl Formal) Backing Films for Evaporated Carbon Substrates

up for carbon evaporation. Pare down one carbon rod using the sharpener to provide a 1-mm diameter, 3-mm long, cylindrical tip. Set this up to make contact in the center of the flat surface of the second carbon rod, which has not been pared down (Fig. 2(*b*)). Center the grids on the screen platform in the evaporator under the a-c glow loop at a distance 100 mm away from the evaporation tip of the carbon rods.

8.2.1.4 Place the bell jar on the evaporator stage and evacuate to an absolute pressure of about 2.7 mPa (2×10^{-5}torr). Apply current to the carbon rods until the tip glows with a red color. Allow the carbon tip to degas at this setting until the pressure remains stable at 2.7 mPa or below.

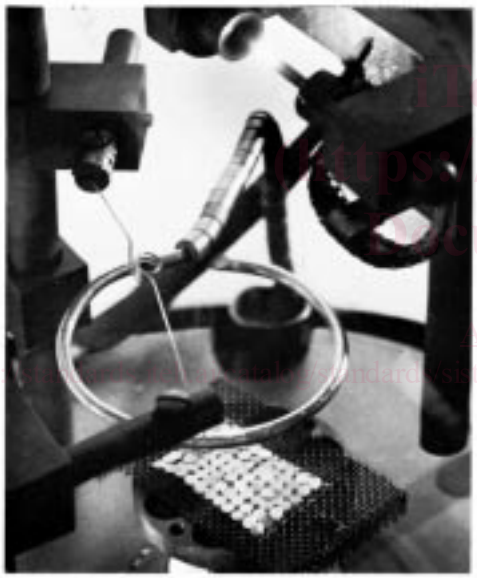
8.2.1.5 Increase the current through the carbon rods until the tip starts to evaporate (Fig. 2(*c*)). Continue to increase the current slowly until the entire tip evaporates. This should take about 30 to 40 s.

8.2.1.6 Turn off the current through the carbon rods and allow the system to cool for about 10 min. Close the valve connecting the oil diffusion pump to the specimen chamber under the evaporator bell jar. Allow air to enter the bell jar until an absolute pressure of 20 Pa (150×10^{-3} torr) is achieved. Activate the a-c glow discharge system at approximately 1500 V. Dim the room lights and inspect the glow pattern. There should be a discernible pinkish glow around the loop, while the region around the specimens (on the grounded stage) is dark (Fig. 2(*d*)). The specimens are in the zone of maximum ion bombardment. Maintain the glow discharge at a pressure of 13 to 20 Pa (100 to 150×10^{-3}torr) for a period of 3 min. Turn off



(*a*) Removal of Poly (Vinyl Formal) Coated Grids from the Water

(*b*) Pared-Down Carbon Rod Tip for Evaporator



(*c*) Evaporation of Carbon on to Poly (Vinyl Formal) Coated Grids

(*d*) a-c Glow Discharge Treatment of Carbon Substrates

FIG. 2 The Preparation of Carbon Substrates for Carbon Black Chloroform Dispersions

the glow discharge and bring the bell jar to atmospheric pressure. Remove the coated specimen grids and store in a dry atmosphere.

NOTE 5—There are other acceptable methods for preparing evaporated carbon substrates. The method described here is necessary for good testing with Dispersion Procedures A and C. The carbon substrates for Dispersion Procedure B are less critical because the carbon black aggregates are suspended in a dried nitrocellulose film at the time they are deposited on the carbon surface. An active hydrophilic carbon film surface is required for the carbon black-chloroform dispersions utilized for Procedures A and C. This is the purpose of the a-c glow discharge treatment. It is further recommended that the air used in the glow treatment have a relatively low moisture content (for room air, a relative humidity no greater than 30 %). Under summertime conditions where the relative humidity of a laboratory often exceeds 30 %, dry air from a tank should be bled into the bell jar for the glow procedure.

For Dispersion Procedure B, it is recommended that the poly (vinyl formal) backing film be removed from the carbon substrates by washing the grids in 1,2-dichloroethane or chloroform. This procedure is also acceptable for Procedures A and C but is not necessary. The deposition of the carbon black-chloroform dispersion removes any large fragments of the poly (vinyl formal) backing film around the edges of the grid, which might contaminate the specimen chamber of the electron microscope over the course of analyzing many specimens. The extra thickness of the backing film has not been found detrimental in obtaining suitable contrast and intensity for electron microscope image analysis procedures.

8.2.2 *Dry Carbon Blacks—Dispersion Procedure A*:

8.2.2.1 Adjust the water level in the tank-type ultrasonic