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NEMA STANDARDS PUBLICATION NO. CB 4

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Semiconductor Graphite

NEMA

NATIONAL ELECTRICAL MANUFACTURERS ASSOCIATION ■ 2101 L STREET, N.W., WASHINGTON, D.C. 20037

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CB 4

SEMICONDUCTOR GRAPHITE

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Foreword

User needs have been considered throughout the development of this Standards Publication.

This Standards Publication, which has been developed by the Carbon Section of the National Electrical Manufacturers Association, will be reviewed periodically for the purpose of assuring compatibility with current OSHA and EPA regulations.

Please address any comments or questions regarding the text of this publication to:

Vice President, Engineering
National Electrical Manufacturers Association
2101 L Street, N.W., Suite 300
Washington, DC 20037
(202) 457-8400

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Scope

This publication defines those physical and chemical properties that are relevant to the material characterization of graphite used in the semiconductor industry. No attempt has been made to incorporate in these standards any application performance standards relative to semiconductor graphite.

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SEMICONDUCTOR GRAPHITE

Section 1
REFERENCED STANDARDS AND DEFINITIONS

1.1 REFERENCED STANDARDS

The following publications are adopted in whole or in part as indicated by reference in this Standards Publication.

National Electrical Manufacturers Association
2101 L Street, N.W.
Washington, DC 20037

ANSI/NEMA CB 1-1984*	*Brushes for Electrical Machines*

American Society for Testing and Materials
1916 Race Street
Philadelphia, PA 19103

ASTM C559-85	*Test Method for Bulk Density by Physical Measurement of Manufactured Carbon and Graphite Articles*
ASTM C561-85	*Test Method for Ash in Graphite*
ASTM C611-84	*Test Method for Electrical Resistivity of Manufactured Carbon and Graphite Articles*
ASTM C561-70 (R 1977)	*Test Method for Flexural Strength of Manufactured Carbon Graphite Articles Using Four-Point Loading at Room Temperature*
ASTM C709-88	*Definitions of Terms Relating to Manufactured Carbon and Graphite*
ASTM C748-83	*Rockwell Hardness Testing of Fine Grained Graphite Materials*
ASTM C886-88	*Scleroscope Hardness Testing of Fine Grained Carbon and Graphite Materials*
ASTM E228-85	*Test Method for Linear Thermal Expansion of Rigid Solids with a Vitreous Silica Dilatometer*

*Also available from the American National Standards Institute, 1430 Broadway, New York, NY 10018

1.2 DEFINITIONS

APPARENT DENSITY
The weight in air of a unit volume including both permeable and impermeable voids present in the material at 25°C.

ASH
The residual product following oxidation of the base carbon as determined by prescribed methods.

CARBON
An element, atomic number 6, symbol C, that exists in several allotropic forms.

COEFFICIENT OF THERMAL EXPANSION (CTE)
The change in length per unit length per unit temperature change. Since the coefficient of thermal expansion of graphite varies with temperature, the temperature range shall be defined.

ELECTRICAL RESISTIVITY
That property of a material which determines its resistance to the flow of an electrical current. Since electrical resistivity of graphite varies with temperature, electrical resistivity shall be reported at a defined temperature.

FLEXURAL STRENGTH
A property of solid material that indicates its ability to withstand a flexural or transverse load.

GRAIN DIRECTION (CONVENTIONALLY MOLDED MATERIAL)
a. With grain: perpendicular to the direction of applied molding force.
b. Against grain: parallel to the direction of applied molding force.

GRAIN DIRECTION (EXTRUDED MATERIAL)
a. With grain: in the direction of extrusion.

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b. Against grain: perpendicular to the direction of extrusion.

GRAIN DIRECTION (ISOSTATICALLY MOLDED MATERIAL)

a. With grain: the direction of the lowest linear thermal expansion.
b. Against grain: the direction of the highest linear thermal expansion.

HARDNESS

The resistance of a material to deformation, particularly permanent deformation, indentation, or scratching.

MANUFACTURED GRAPHITE

A bonded granular carbon with a matrix that has been subjected to a temperature typically in excess of 2400°C and is thermally stable below that temperature.

MATERIAL GRADE DESIGNATION

The designation given a material by a manufacturer such that it is always produced to the same specifications established by the manufacturer.

MAXIMUM GRAIN SIZE

The largest particle of the granular component as determined by its major dimension. In practice, commonly given by the nominal opening of the coarsest screen used to prepare the initial raw materials in making the product and through which substantially all initial raw material will pass or by other measuring techniques, such as the Fisher subsieve, Coulter counter, and such.

OXIDATION OF CARBON

The chemical combination of carbon with oxygen or oxygen containing compounds.

PURITY

A quality of graphite based on its ash content such that higher purity relates to lower ash.

PURIFIED GRAPHITE

Graphite that has been subjected to processes specifically aimed at reduction of its ash content.

PYROLYTIC GRAPHITE

Carbon material resulting from the thermal decomposition of certain hydrocarbon gasses.

SILICON-CARBIDE COATED GRAPHITE

A graphite that has a chemical vapor deposited (CVD) surface layer of silicon carbide resulting from high temperature reaction of silicon and carbon containing gases on a graphite substrate.

SILICON CARBIDE CONVERTED GRAPHITE (CVR)

A graphite with a surface that has been converted to silicon carbide by silicon vapor reaction (CVR) at elevated temperature.

VITREOUS (GLASSY) CARBON

A carbon material resulting from the thermal decomposition of certain resins.

NEMA Standard 11-16-1989.

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Section 2
GRAPHITE PROPERTIES

2.1 CLASSIFICATION OF GRAIN SIZE

Semiconductor graphite material can be classified by grain size as follows:

a. Ultrafine (or micrograin graphite) – less than 10 microns maximum grain size;
b. Superfine – between 10 and 50 microns maximum grain size;
c. Fine – greater than 50 but less than or equal to 100 microns maximum grain size; or
d. Medium – greater than 100 microns maximum grain size.

The above classifications are commonly based on size measuring techniques of the initial raw materials. It should be noted that the measuring techniques involved are not applicable to the finished product.

Authorized Engineering Information 11-16-1989.

2.2 CLASSIFICATION OF PURITY

Graphites are typically available in the following classifications of purity:

> 1,000 PPM ash
< 1,000 PPM ash
< 500 PPM ash
< 100 PPM ash
< 20 PPM ash

Authorized Engineering Information 11-16-1989.

2.3 PROCEDURES FOR MATERIAL TESTING

2.3.1 Flexural Strength

The flexural strength shall be determined in accordance with ASTM C651 or in accordance with the procedure specified in 4.7 of ANSI/NEMA CB 1.

Test data derived from use of these test procedures shall identify the method used.

NEMA Standard 11-16-1989.

2.3.2 Hardness

Graphite shall be tested in accordance with either the scleroscope hardness method specified in 3.2 of ANSI/NEMA CB 1 or ASTM C886 or the indentation hardness method specified in 3.3 of ANSI/NEMA CB 1 or ASTM C748.

Test data shall identify the method used.

NEMA Standard 11-16-1989.

2.3.3 Electrical Resistivity

The electrical resistivity of semiconductor graphite shall be determined in accordance with the voltmeter-ammeter method specified in 3.4 of ANSI/NEMA CB 1, or ASTM C611.

NEMA Standard 11-16-1989.

2.3.4 Density (Apparent)

The apparent density of graphite shall be determined using the immersion method specified in 3.6.3 of ANSI/NEMA CB 1 or the weight in air method given in ASTM C559.

Test data shall identify the method used.

NEMA Standard 11-16-1989.

2.3.5 Ash Content

Ash content shall be determined using the procedure specified in 3.8 of ANSI/NEMA CB 1 or ASTM C561.

NEMA Standard 11-16-1989.

To determine ash level of purified graphite the procedures described in the ASTM Method C561 may need to be modified to provide accuracy at very low levels, i.e., less than 100 PPM. Improved weighing apparatus or increased sample size are the common modifications used by the graphite industry.

Authorized Engineering Information 11-16-1989.

2.3.6 Coefficient of Thermal Expansion

The coefficient of thermal expansion (CTE) can be determined by using the procedures specified in ASTM E228.

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Section 3
GENERAL

Section 3 is classified as NEMA Authorized Engineering Information.

3.1 HANDLING OF PURIFIED GRAPHITE

In order to prevent contamination of purified graphite, certain precautions should be taken as follows:

a. Purified graphite should be stored in a protective envelope whenever possible.
b. Cleanliness should be insured for everything coming in contact with the purified graphite, e.g., clamps, lifting tools, gloves, and such. Precaution should be taken so that material transfer does not occur from handling equipment to the purified graphite surface.
c. In certain cases, a clean room atmosphere and/or positive pressure work area may be required.

3.2 DUST REMOVAL

During the machining of graphite, some of the dust generated becomes mechanically enmeshed within the surface porosity of the parent graphite. This material should be removed so it does not act later as a contaminant. This is usually accomplished by ultrasonic cleaning followed by packaging the graphite in a protective envelope.

3.3 OUTGASSING

A large number of chemically active edge sites are contained in a typical polycrystalline graphite. These edge carbons have a marked affinity for the chemisorption of various gaseous molecules (e.g., CO, CO_2, N_2, H_2, H_2O).

In a high temperature vacuum environment, these gases have a tendency to desorb and thereby become a potential source of contamination.

In those applications where such contaminants are not permitted, the graphite should be subjected to a high temperature vacuum degassing cycle prior to use. Such a cycle typically calls for degassing the graphite at a temperature greater than 2000°C under a pressure of less than 10 torr followed by backfilling with a non-contaminating gas such as argon during the cool-down cycle.

3.4 OXIDATION

The oxidation of graphite is a well-known phenomenon. This reaction is often the life-limiting criteria in a system that involves the use of graphite components at elevated temperatures, particularly above 500°C.

The primary oxidizing agents, given in the order of their reactivity, are O_2, CO_2, and H_2O. It should be noted that the rate-determining step in the oxidation of graphite is usually the catalytic activity of metal impurities. The removal of these impurities will often reduce the reaction rate to an acceptable level. If this is not sufficient, one should either: (1) prevent the oxidant from contacting the graphite (e.g. by using a SiC barrier coating) or (2) eliminate the oxidant from the system (i.e., by using a protective atmosphere or a vacuum).

3.5 PURIFICATION

The purification of graphite is often performed in a high temperature, halogen gas environment to remove ash components inherent in the graphite. For semiconductor applications, gas purification is generally performed after machining when ash or purity levels of less than 20 PPM are required. Suppliers of semiconductor graphite will provide certification of ash level and specific spectral analyses by element when requested.

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NEMA STANDARDIZATION

The purpose of NEMA Standards, their classification and status, are set forth in certain clauses of the NEMA *Standardization Policies and Procedures* manual and are referenced below.

Purpose of Standards

National Electrical Manufacturers Association standards are adopted in the public interest and are designed to eliminate misunderstandings between the manufacturer and the purchaser and to assist purchasers in selecting and obtaining the proper product for their particular needs. Existence of a National Electrical Manufacturers Association standard does not in any respect preclude any member or nonmember from manufacturing or selling products not conforming to the standard. *(Standardization Policies and Procedures, p. 1)*

Definition of a Standard

A standard of the National Electrical Manufacturers Association defines a product, process, or procedure with reference to one or more of the following: nomenclature, composition, construction, dimensions, tolerances, safety, operating characteristics, performance, rating, testing, and the service for which they are designed.

(Standardization Policies and Procedures, p. 2)

Dimensions

Where dimensions are given for interchangeability purposes, alternate dimensions satisfying the other provisions of the Standards Publication may be capable of otherwise equivalent performance.

(Standardization Policies and Procedures, p.8)

Categories of Standards

National Electrical Manufacturers Association Standards are of two classes:

1. NEMA Standard, which relates to a product, process, or procedure commercially standardized and subject to repetitive manufacture, which standard has been approved by at least 90 percent of the members of the Subdivision eligible to vote thereon;
2. Suggested Standard for Future Design, which may not have been regularly applied to a commercial product, but which suggests a sound engineering approach to future development, which standard has been approved by at least two-thirds of the members of the Subdivision eligible to vote thereon.

(Standardization Policies and Procedures, pp. 7 & 16)

Authorized Engineering Information

Authorized Engineering Information consists of explanatory data and other engineering information of an informative character not falling within the classification of NEMA Standard or Suggested Standard for Future Design, which standard has been approved by at least two-thirds of the members of the Subdivision eligible to vote on the standard.

(Standardization Policies and Procedures, pp. 7 & 16)

Official Standards Proposal

An Official Standards Proposal is an official draft of a proposed standard which is formally recommended to an outside organization(s) for consideration, comment, and/or approval, and which has been approved by at least 90 percent of the members of the Subdivision eligible to vote thereon.

(Standardization Policies and Procedures, pp. 7 & 16)

Identification of Status

Standards in NEMA Standards Publications are identified in the foreword or following each standard as "NEMA Standard" or "Suggested Standard for Future Design." These indicate the status of the standard. These words are followed by a date which indicates when the standard was adopted in its present form by the Association.

The material identified as "Authorized Engineering Information" and "Official Standards Proposal" is designated similarly.

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CARBON SECTION
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MEMBER COMPANIES

Carbone USA Corporation
Boonton, NJ 07005

Carbon Engineering Corporation
Slinger, WI 53086

Carbon Products Operations, Inc.
East Stroudsburg, PA 18301

The Carbon/Graphite Group, Inc.
Pittsburgh, PA 15222

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