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An American National Standard

Standard Specification for Nuclear Graphite Suitable for Components Subjected to Low Neutron Irradiation Dose[1]

This standard is issued under the fixed designation D7301; 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 specification covers the classification, processing, and properties of nuclear grade graphite billets with dimensions sufficient to meet the designer's requirements for reflector blocks and core support structures, in a high temperature gas cooled reactor. The graphite classes specified here would be suitable for reactor core applications where neutron irradiation induced dimensional changes are not a significant design consideration.

1.2 The purpose of this specification is to document the minimum acceptable properties and levels of quality assurance and traceability for nuclear grade graphite suitable for components subjected to low irradiation dose. Nuclear graphites meeting the requirements of Specification D7219 are also suitable for components subjected to low neutron irradiation dose.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C559 Test Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles

C709 Terminology Relating to Manufactured Carbon and Graphite

C781 Practice for Testing Graphite and Boronated Graphite Materials for High-Temperature Gas-Cooled Nuclear Reactor Components

C838 Test Method for Bulk Density of As-Manufactured Carbon and Graphite Shapes

C1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials

D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis

D2638 Test Method for Real Density of Calcined Petroleum Coke by Helium Pycnometer

D7219 Specification for Isotropic and Near-isotropic Nuclear Graphites

IEEE/ASTM SI 10 American National Standard for Use of the International System of Units (SI): The Modern Metric System

2.2 *ASME Standards:*[3]

NQA-1 Quality Assurance Program Requirements for Nuclear Facilities

3. Terminology

3.1 *Definitions*—Definitions relating to this specification are given in Terminology C709. See Table 1.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *anistropic nuclear graphite, n*—graphite in which the isotropy ratio based on the coefficient of thermal expansion is greater than 1.15.

3.2.2 *baking/re-baking charge, n*—number of billets in a baking/re-baking furnace run.

3.2.3 *bulk density, n*—mass of a unit volume of material including both permeable and impermeable voids.

3.2.4 *extrusion forming lot, n*—number of billets of the same size extruded in an uninterrupted sequence.

3.2.5 *green batch, n*—mass of coke, recycle green mix, recycle graphite, and pitch that is required to produce a forming lot.

3.2.6 *green mix, n*—percentage of mix formulation, pitch and additives required for the forming lot, which is processed and ready to be formed.

3.2.7 *graphite billet, n*—extruded, molded, or iso-molded graphite artifact with dimensions sufficient to meet the designer's requirements for reactor components.

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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.

[3] Available from American Society of Mechanical Engineers (ASME), ASME International Headquarters, Three Park Ave., New York, NY 10016-5990, http://www.asme.org.

TABLE 1 ASTM Graphite Grain Size Definitions from Terminology C709

Graphite Designation	Definition of Grains in the Starting Mix that are:[A]
Medium Grained	Generally < 4 mm[B]
Fine Grained	Generally < 100 µm
Superfine Grained	Generally < 50 µm
Ultrafine Grained	Generally < 10 µm
Microfine Grained	Generally < 2 µm

[A] Grain size as defined in Terminology C709.
[B] For Nuclear graphite, the maximum grain size is 1.68 mm in accordance with 4.2.1.6.

3.2.8 *graphite grade, n*—designation given to a material by a manufacturer such that it is always reproduced to the same specification and from the same raw materials and mix formulation.

3.2.9 *graphitizing furnace run, n*—total number of billets graphitized together in one graphitization furnace.

3.2.10 *graphitization charge, n*—total number of billets graphitized together in one graphitization furnace.

3.2.11 *high purity nuclear graphite, n*—nuclear graphite with an Equivalent Boron Content less than 2 ppm.

3.2.12 *impregnation charge, n*—number of billets in an autoclave cycle.

3.2.13 *low purity nuclear graphite, n*—nuclear graphite with an Equivalent Boron Content greater than 2 ppm but less than 10 ppm.

3.2.14 *mix formulation, n*—percentages of each specifically sized filler used to manufacture a graphite grade.

3.2.15 *molding forming lot, n*—number of billets molded from a molding powder lot.

3.2.16 *molding powder lot, n*—sufficient quantity of re-milled and blended green batch produced from an uninterrupted flow of raw materials, or produced in a sequence of identical materials batches, to produce a molding forming lot.

3.2.17 *nuclear graphite class, n*—designation of a nuclear graphite based upon its forming method, isotropy, purity and density (see Table 2).

3.2.18 *production lot, n*—specified number of billets made in accordance with this specification and additional requirements determined by the purchaser.

3.2.19 *purification charge, n*—number of billets in a purification run.

3.2.20 *recycle green mix, n*—ground non-baked billets or non-used green mix manufactured in compliance with the mix formulation specified here.

4. Materials and Manufacture

4.1 *Nuclear Graphite Classes*—See Table 2.

4.2 *Raw Materials:*

4.2.1 *Fillers:*

4.2.1.1 The filler shall consist of a coke derived from a petroleum oil or coal tar.

4.2.1.2 The coke shall have a coefficient of linear thermal expansion (CTE), determined in accordance with Practice C781 and measured over the temperature range 25 °C to 500 °C, less than 5.5×10^{-6}°C^{-1}.

4.2.1.3 The coke shall be sampled and distributed as described in Table 3.

4.2.1.4 Graphite manufactured in compliance with this specification but failing to meet the property requirements of Sections 5 – 7 may be used as recycle material in the mix formulation.

4.2.1.5 Recycle green mix manufactured from raw materials in compliance with this specification may be used in the mix formulation.

4.2.1.6 The maximum filler particle size used in the mix formulation shall be 1.68 mm.

4.3 *Binder*—The binder shall consist of coal tar pitch of the same grade from the same manufacturer. The specific binder(s) used shall be identified to the purchaser and be traceable through the forming lot.

4.4 *Impregnant*—The impregnant shall consist of a petroleum or coal tar pitch of the same grade from the same manufacturer. The specific impregnant(s) used shall be identified to the purchaser and be traceable through the impregnation steps.

4.5 *Manufacturing or Processing Additives*—Additives (for example, extrusion aids) may be used to improve the processing, quality and properties of the product, but only with the consent and approval of the purchaser, and they must be traceable through the forming lot.

4.6 *Manufacture:*

TABLE 2 ASTM Standard Classes of Nuclear Graphite

Class[A]	CTE Isotropy Ratio[B] (α_{AG}/α_{AG})	Purity: Ash Content,[C] ppm (max)	Purity: Boron Equivalent,[D] ppm (max)	Density, g/cm^3 (min)	Class Designation
Isomolded, anisotropic-HP	>1.15	300	2	1.7	IAHP
Isomolded, anisotropic-LP	>1.15	1000	10	1.7	IALP
Extruded, anisotropic-HP	>1.15	300	2	1.7	EAHP
Extruded, anisotropic-LP	>1.15	1000	10	1.7	EALP
Molded, anisotropic-HP	>1.15	300	2	1.7	MAHP
Molded, anisotropic-LP	>1.15	1000	10	1.7	MALP

[A] These classes may be further modified by the grain size as defined in Terminology C709.
[B] Determined in accordance with Practice C781.
[C] Determined in accordance with Test Method C559.
[D] Determined in accordance with Practice C1233.

TABLE 3 Inspection Sampling and Testing of Filler Cokes

Inspection Plan	Sampling Procedure	Tests and Test Methods
A representative sample of the coke shall be taken prior to the mixing step of manufacture	Sample in accordance with Practice D346 1. A sufficient sample for preparation of CTE test specimens 2. A sufficient sample will be taken for additional testing. This sample shall be retained for a period specified by the graphite purchaser	The procedure in Practice C781 shall be used to prepare test specimens for the measurement of coke CTE Measure the coke real density in accordance with Test Method D2638

4.6.1 *Formulation*—The mix formulation (as defined in 3.2.14) and recycle green mix fraction (as defined in 3.2.20) in the filler shall be recorded. This information shall be reported to the purchaser if requested.

4.6.2 *Forming*—The green mix may be formed by extrusion, molding (including vibrationally molding), or iso-molding.

4.6.3 *Graphitization Temperature*—The graphitization temperature shall be determined on each billet using the procedure described in Practice C781. Each billet tested in accordance with Practice C781 shall have a Specific Electrical Resistivity (SER) corresponding to a graphitization temperature of at least 2700°C.

5. Chemical Properties

5.1 Each graphite production lot shall be sampled in accordance with Section 10. The chemical impurities to be measured shall be agreed upon between the supplier and the purchaser. The minimum list of elements to be measured and used for the EBC calculation shall be B, Cd, Dy, Eu, Gd, and Sm.

5.2 The boron equivalent shall be calculated in accordance with Practice C1233. The acceptance limits for the boron equivalent as well as for ash content are given in Table 2.

5.3 Table X1.1 contains a list of chemical impurities that are typically measured depending on end-use requirements. The impurities are categorized as neutron absorbing impurities, oxidation promoting catalysts, activation relevant impurities, metallic corrosion relevant impurities, and fissile/fissionable elements.

6. Physical and Mechanical Properties

6.1 Each graphite production lot shall be sampled in accordance with Section 10 and shall conform to the requirements for physical properties prescribed in Table 2 and Table 4 for the appropriate nuclear graphite class, and to the additional requirements of the purchaser.

6.2 The bulk density of each graphite billet shall be measured as described in Test Method C838.

7. Other Requirements

7.1 The graphitized billets shall be handled and stored such that they are protected from contaminants other than ambient air.

7.2 Each graphite billet shall be marked with a unique billet identification number. Each billet shall be traceable through these identifying numbers to each of the following:

7.2.1 Mix formulation,

7.2.2 Coke batch,

7.2.3 Recycle graphite batch,

7.2.4 Forming lot,

7.2.5 Molding powder lot,

7.2.6 Baking charge,

7.2.7 Impregnant charge,

7.2.8 Graphitization furnace run,

7.2.9 Position of billet in graphitization furnace,

7.2.10 Purification step (if performed),

7.2.11 Binder pitch,

7.2.12 Impregnant pitch, and

7.2.13 Additives used (if any).

8. Dimensions

8.1 Graphite billet dimensions are typically 0.4 m to 0.6 m in diameter (extruded) or thickness (molded/extruded) of 0.6 m by 0.6 m cross-section (iso-molded) and 0.75 m to 3.0 m in length.

TABLE 4 Physical and Mechanical Properties for Nuclear Graphite

Class[B]	Coefficient of Thermal Expansion (25 °C to 500 °C), WG, $°C^{-1}$	Thermal Conductivity at 25 °C, AG $Wm^{-1}K^{-1}$ (min)	Strength,[A] WG, MPa, min			Dynamic Elastic Modulus, WG GPa (min)
			Tensile	Flexural	Compressive	
Practice[C]	C781	C781	C781	C781	C781	C781
IAHP	Less than 5.5×10^{-6}	80	20	30	55	8
IALP	Less than 5.5×10^{-6}	80	20	30	55	8
EAHP	Less than 5.5×10^{-6}	100	13	20	43	8
EALP	Less than 5.5×10^{-6}	100	13	20	43	8
MAHP	Less than 5.5×10^{-6}	90	8	11	24	4
MALP	Less than 5.5×10^{-6}	90	8	11	24	4

[A] At least one of the three strength measurements should be selected for production lot acceptance in agreement with the supplier and the purchaser.
[B] WG = With Grain; AG = Against Grain.
[C] Equivalent practices may be used by manufacturers based outside the United States.

9. Workmanship, Finish, and Appearance

9.1 Graphitized billets shall be brushed clean after removal from the graphitization furnace.

10. Sampling and Cutting

10.1 A statistical sampling plan shall be developed by the supplier and agreed upon with the purchaser. The plan shall describe the number of graphite billets to be sampled and the frequency of sampling. The following minimum sampling frequencies are recommended per production lot depending on the number of billets per production lot.

10.1.1 Sample 4 billets for each production lot containing 10 or fewer billets.

10.1.2 Sample one additional billet for every 5 additional billets per production lot up to 50 billets.

10.1.3 For production lots exceeding 50 billets the additional sampling requirements should be agreed upon by the supplier and the purchaser.

10.1.4 During production the sampling plan may be re-evaluated based on statistical analysis of the production data. Any revised sampling plan must be agreed upon between the supplier and the purchaser.

10.2 A cutting plan shall be agreed upon by the purchaser and manufacturer. The cutting plan shall describe the type, location, number, orientation of the test specimens, and any required archive specimens needed for property determinations as set forth in Sections 5 – 7 of this specification. The cutting plan shall reflect property gradients and anisotropy introduced by forming and processing. In addition the number of each type of specimen defined by the cutting plan shall be sufficient to yield statistically significant data.

11. Finished Inspection

11.1 Graphite billets shall be visually inspected for external flaws. The allowable size, type, and number of flaws shall be defined in the agreement between the purchaser and the manufacturer and be described in the purchase specification.

11.2 It is recommended that all graphite billets are non-destructively tested to screen for internal defects. The allowable size, type, and number of internal flaws should be defined in the agreement between the purchaser and the manufacturer and be described in the purchase specification.

12. Rejection and Rework

12.1 Graphite billets failing on chemical purity (see Section 5) may be purified/re-purified and subjected to retest.

12.2 Graphite billets failing on SER (4.6.3) may be re-graphitized and subjected to retest.

12.3 All other billets failing to meet the requirement of Sections 5 – 7 of this specification may be used as recycle graphite in accordance with 4.2.1.4.

13. Certification

13.1 The manufacturer shall certify that the graphite meets the requirements of this specification and the purchase specification.

14. Product Marking

14.1 Each billet shall be marked with a permanent unique number, which shall be traceable to the production history as specified in 7.2.

15. Packaging and Storage

15.1 Packaging of the finished billet shall be in accordance with the purchase specification.

15.2 Storage of the finished billets prior to shipping shall be such that no damage is incurred.

16. Quality Assurance

16.1 The manufacturer of nuclear graphite furnished under this specification shall comply with the applicable quality assurance requirements of the specific version of ASME NQA-1 as identified by the purchase specification. The purchase specification may require application of quality assurance requirements other than ASME NQA-1.

17. Keywords

17.1 chemical properties; mechanical properties; nuclear graphite; physical properties; processing

APPENDIX

(Nonmandatory Information)

X1. IMPURITIES

X1.1 The control of impurities in nuclear graphite depends on the specific end-use requirements. Table X1.1 categorizes typical chemical impurities found in nuclear graphite that contribute to specific effects, for example, neutron absorption. The contributing effect of the impurity element depends on both the nature of the impurity and its concentration in the graphite. For example, boron and gadolinium have a high neutron absorption cross-section, but in the high purity nuclear graphite the quantities are low. However, an element such as iron that has a low absorption cross-section can typically be an order of magnitude higher in concentration and thus influence the overall Equivalent Boron Content, more so in the low purity nuclear graphite. It is therefore recommended that the supplier provide chemical impurity data on the nuclear graphite to be supplied from which the key impurities to be measured can be agreed upon with the purchaser.

TABLE X1.1 Impurity Categories for Nuclear Graphites[A]

Neutron Absorbing Impurities	Oxidation Promoting Catalysts	Activation Relevant Impurities	Metallic Corrosion Relevant Impurities	Fissile/Fissionable Elements
Boron	Aluminum	Antimony (Sb)	Chlorine	Thorium
Cadmium	Barium	Cadmium	Mercury	Uranium
Chlorine	Calcium	Cesium	Sulfur	
Dysprosium	Copper	Chlorine[B]		
Europium	Iron	Cobalt[A]		
Gadolinium	Lead	Europium		
Gold	Magnesium	Gadolidium		
Indium	Manganese	Iron		
Iron	Nickel[C]	Lithium[D]		
Mercury	Potassium	Molybdenum		
Nickel	Silicon	Nickel		
Rhenium	Silver	Nitrogen[E]		
Rhodium	Sodium	Scandium		
Samarium	Titanium	Silver		
Titanium		Tantalum		
Vanadium		Terbium		
		Thorium		
		Uranium		
		Zinc		

[A] Problem isotope is ^{60}Co, strong gamma emitter, long half-life.
[B] Active isotope is ^{36}Cl (gamma emitter with extremely long half-life.
[C] Source of ^{60}Co.
[D] Primary source of tritium ^{3}H (beta emitter).
[E] Primary source of ^{14}C, beta emitter with long half-life.