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An American National Standard

Standard Specification for Nuclear Graphite Suitable for Components Subjected to Low Neutron Irradiation Dose¹

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.

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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.

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TABLE 1 ASTM Graphite Grain Size Definitions from Terminology

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	Graphite	Definition of Grains in
	Designation	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
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^A Grain size as defined in Terminology C709.

^B For Nuclear graphite, the maximum grain size is 1.68 mm <u>1.68 mm</u> in accordance with 4.2.1.6.

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.

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.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 2 ppm but less than 10 ppm. 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). $\underline{\text{ASTM D7301-11}(2015)}$

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:

TABLE 2 ASTM Standard Classes of Nuclear Graphite

Purity						
Class ^A	CTE Isotropy Ratio ^B $(\alpha_{AG}/\alpha_{AG})$	Ash Content, ^C ppm (max)	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.

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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 2525 °C to 500 °C, less than $5.5 \times 10^{-6} \text{ °C}^{-1}$.

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:

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

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,

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	The procedure in Practice C781 shall be used to prepare test specimens for the measurement of coke CTE				
	 A sufficient sample will be taken for additional testing. This sample shall be retained for a period specified by the graphite purchaser 	Measure the coke real density in accordance with Test Method D2638				

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