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Standard Guide for Development of Specifications for Fiber Reinforced Silicon Carbide-Silicon Carbide Composite Structures for Nuclear Applications[1]

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1. Scope

1.1 This document is a guide to preparing material specifications for silicon carbide fiber/silicon carbide matrix (SiC-SiC) composite structures (flat plates, rectangular bars, round rods, and tubes) manufactured specifically for structural components and for fuel cladding in nuclear reactor core applications. The SiC-SiC composites consist of silicon carbide fibers in a silicon carbide matrix produced by liquid infiltration/pyrolysis and/or by chemical vapor infiltration.

1.2 This guide provides direction and guidance for the development of a material specification for a specific SiC-SiC composite component or product for nuclear reactor applications. The guide considers composite constituents and structure, physical and chemical properties, mechanical properties, thermal properties, performance durability, methods of testing, materials and fabrication processing, and quality assurance. The SiC-SiC composite materials considered here would be suitable for nuclear reactor core applications where neutron irradiation-induced damage and dimensional changes are significant design considerations. (**1-8**)[2]

1.3 The component material specification is to be developed by the designer/purchaser/user. The designer/purchaser/user shall define and specify in detail any and all application-specific requirements for design, manufacturing, performance, and quality assurance of the ceramic composite component. Additional specification items for a specific component, beyond those listed in this guide, may be required based on intended use, such as geometric tolerances, permeability, bonding, sealing, attachment, and system integration.

1.4 This guide is specifically focused on SiC-SiC composite components and structures with flat plate, solid rectangular bar, solid round rod, and tubular geometries.

1.5 This guide may also be applicable to the development of specifications for SiC-SiC composites used for other structural applications, discounting the nuclear-specific chemical purity and irradiation behavior factors.

1.6 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.7 *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]

C242 Terminology of Ceramic Whitewares and Related Products

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

C577 Test Method for Permeability of Refractories

C611 Test Method for Electrical Resistivity of Manufactured Carbon and Graphite Articles at Room Temperature

C625 Practice for Reporting Irradiation Results on Graphite

C714 Test Method for Thermal Diffusivity of Carbon and Graphite by Thermal Pulse Method

C769 Test Method for Sonic Velocity in Manufactured Carbon and Graphite Materials for Use in Obtaining Young's Modulus

C816 Test Method for Sulfur in Graphite by Combustion-Iodometric Titration Method

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

C1039 Test Methods for Apparent Porosity, Apparent Specific Gravity, and Bulk Density of Graphite Electrodes

C1145 Terminology of Advanced Ceramics

[1] This guide is under the jurisdiction of ASTM Committee C28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.07 on Ceramic Matrix Composites.

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[2] The boldface numbers in parentheses refer to the list of references at the end of this standard.

[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.

C1179 Test Method for Oxidation Mass Loss of Manufactured Carbon and Graphite Materials in Air
C1198 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Sonic Resonance
C1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials
C1239 Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics
C1259 Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Impulse Excitation of Vibration
C1274 Test Method for Advanced Ceramic Specific Surface Area by Physical Adsorption
C1275 Test Method for Monotonic Tensile Behavior of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross-Section Test Specimens at Ambient Temperature
C1291 Test Method for Elevated Temperature Tensile Creep Strain, Creep Strain Rate, and Creep Time-to-Failure for Advanced Monolithic Ceramics
C1292 Test Method for Shear Strength of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperatures
C1337 Test Method for Creep and Creep Rupture of Continuous Fiber-Reinforced Advanced Ceramics Under Tensile Loading at Elevated Temperatures
C1341 Test Method for Flexural Properties of Continuous Fiber-Reinforced Advanced Ceramic Composites
C1358 Test Method for Monotonic Compressive Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross-Section Test Specimens at Ambient Temperatures
C1359 Test Method for Monotonic Tensile Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics With Solid Rectangular Cross-Section Test Specimens at Elevated Temperatures
C1360 Practice for Constant-Amplitude, Axial, Tension-Tension Cyclic Fatigue of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperatures
C1425 Test Method for Interlaminar Shear Strength of 1–D and 2–D Continuous Fiber-Reinforced Advanced Ceramics at Elevated Temperatures
C1468 Test Method for Transthickness Tensile Strength of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperature
C1470 Guide for Testing the Thermal Properties of Advanced Ceramics
C1525 Test Method for Determination of Thermal Shock Resistance for Advanced Ceramics by Water Quenching
C1557 Test Method for Tensile Strength and Young's Modulus of Fibers
C1683 Practice for Size Scaling of Tensile Strengths Using Weibull Statistics for Advanced Ceramics
C1773 Test Method for Monotonic Axial Tensile Behavior of Continuous Fiber-Reinforced Advanced Ceramic Tubular Test Specimens at Ambient Temperature
D2766 Test Method for Specific Heat of Liquids and Solids
D3171 Test Methods for Constituent Content of Composite Materials
D3529/D3529M Test Method for Matrix Solids Content and Matrix Content of Composite Prepreg
D3800 Test Method for Density of High-Modulus Fibers
D3878 Terminology for Composite Materials
D4018 Test Methods for Properties of Continuous Filament Carbon and Graphite Fiber Tows
D4284 Test Method for Determining Pore Volume Distribution of Catalysts and Catalyst Carriers by Mercury Intrusion Porosimetry
D4850 Terminology Relating to Fabrics and Fabric Test Methods
D5528 Test Method for Mode I Interlaminar Fracture Toughness of Unidirectional Fiber-Reinforced Polymer Matrix Composites
D5600 Test Method for Trace Metals in Petroleum Coke by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)
D5766 Test Method for Open-Hole Tensile Strength of Polymer Matrix Composite Laminates
D5961 Test Method for Bearing Response of Polymer Matrix Composite Laminates
D6484 Test Method for Open-Hole Compressive Strength of Polymer Matrix Composite Laminates
D6507 Practice for Fiber Reinforcement Orientation Codes for Composite Materials
D6671 Test Method for Mixed Mode I-Mode II Interlaminar Fracture Toughness of Unidirectional Fiber Reinforced Polymer Matrix Composites
D7136 Test Method for Measuring the Damage Resistance of a Fiber-Reinforced Polymer Matrix Composite to a Drop-Weight Impact Event
D7137 Test Method for Compressive Residual Strength Properties of Damaged Polymer Matrix Composite Plates
D7219 Specification for Isotropic and Near-isotropic Nuclear Graphites
D7542 Test Method for Air Oxidation of Carbon and Graphite in the Kinetic Regime
E6 Terminology Relating to Methods of Mechanical Testing
E111 Test Method for Young's Modulus, Tangent Modulus, and Chord Modulus
E132 Test Method for Poisson's Ratio at Room Temperature
E143 Test Method for Shear Modulus at Room Temperature
E228 Test Method for Linear Thermal Expansion of Solid Materials With a Push-Rod Dilatometer
E261 Practice for Determining Neutron Fluence, Fluence Rate, and Spectra by Radioactivation Techniques
E289 Test Method for Linear Thermal Expansion of Rigid Solids with Interferometry
E408 Test Methods for Total Normal Emittance of Surfaces Using Inspection-Meter Techniques
E423 Test Method for Normal Spectral Emittance at Elevated Temperatures of Nonconducting Specimens
E1269 Test Method for Determining Specific Heat Capacity by Differential Scanning Calorimetry

E1309 Guide for Identification of Fiber-Reinforced Polymer-Matrix Composite Materials in Databases (Withdrawn 2015)[4]
E1461 Test Method for Thermal Diffusivity by the Flash Method
E1922 Test Method for Translaminar Fracture Toughness of Laminated and Pultruded Polymer Matrix Composite Materials
E2586 Practice for Calculating and Using Basic Statistics

2.2 *Non-ASTM Standards:*
CMH-17, Volume 5 Composite Materials Handbook (CMC Handbook)
ASME B46.1-2009 Surface Texture (Surface Roughness, Waviness, and Lay)[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *General*—Many of the terms in this guide for specifications are defined in the terminology standards for ceramic whitewares (C242), advanced ceramics (C1145), composite materials (D3878), fabrics and test methods (D4850), and mechanical testing (E6).

3.1.2 *apparent porosity, n*—the volume fraction of all pores, voids, and channels within a solid mass that are interconnected with each other and communicate with the external surface, and thus are measurable by gas or liquid penetration. (Synonym – open porosity) **C242**

3.1.3 *braided fabric, n*—a woven structure produced by interlacing three or more ends of yarns in a manner such that the paths of the yarns are diagonal to the vertical axis of the fabric. **D4850**

3.1.3.1 *Discussion*—Braided structures can have 2D or 3D architectures.

3.1.4 *bulk density, n*—the mass of a unit volume of material including both permeable and impermeable voids. **D7219**

3.1.5 *ceramic matrix composite, n*—a material consisting of two or more materials (insoluble in one another), in which the major, continuous component (matrix component) is a ceramic, while the secondary component(s) (reinforcing component) may be ceramic, glass-ceramic, glass, metal or organic in nature. These components are combined on a macroscale to form a useful engineering material possessing certain properties or behavior not possessed by the individual constituents. **C1145**

3.1.6 *fabric, n—in textiles*, a planar structure consisting of yarns or fibers. **D4850**

3.1.7 *fiber, n*—a fibrous form of matter with an aspect ratio >10 and an effective diameter <1 mm. (Synonym – filament) **D3878**

3.1.7.1 *Discussion*—A fiber/filament forms the basic element of fabrics and other textile structures.

3.1.8 *fiber areal weight, n*—the mass per unit area of the fibrous reinforcement of a composite material. **D3529/D3529M**

3.1.9 *fiber content/fraction (volume or weight), n*—the amount of fiber present in a composite, expressed either as a percent by weight or a percent by volume. **D3878**

3.1.10 *fiber preform, n*—a preshaped fibrous reinforcement, normally without matrix, but often containing a binder to facilitate manufacture, formed by distribution/weaving of fibers to the approximate contour and thickness of the finished part. **D3878**

3.1.11 *fill, n—in a woven fabric*, the yarn running from selvage to selvage at right angles to the warp. **D3878**

3.1.12 *hybrid, n*—(for composite materials) containing at least two distinct types of matrix or reinforcement. Each matrix or reinforcement type can be distinct because of its a) physical or mechanical properties, or both, b) material form, or c) chemical composition. **D3878**

3.1.13 *injection molding, n—in composite fabrication*, the process of forcing liquid polymer under pressure into a closed mold that contains a fiber preform.

3.1.14 *knitted fabric, n*—a fiber structure produced by interlooping one or more ends of yarn or comparable material. **D4850**

3.1.15 *laminate, n*—any fiber- or fabric-reinforced composite consisting of laminae (plies) with one or more orientations with respect to some reference direction. **D3878**

3.1.16 *lay-up, n*—a process or fabrication involving the placement of successive layers of materials in specified sequence and orientation. **E1309, D6507**

3.1.17 *matrix, n*—the continuous constituent of a composite material, which surrounds or engulfs the embedded reinforcement in the composite and acts as the load transfer mechanism between the discrete reinforcement elements. **D3878**

3.1.18 *matrix content, n*—the amount of matrix present in a composite expressed either as a percent by weight or a percent by volume. **D3878**

3.1.19 *ply, n—in 2D laminar composites*, the constituent single layer as used in fabricating, or occurring within, a composite structure. **D3878**

3.1.20 *prepreg, n*—the admixture of fibrous reinforcement and polymeric matrix used to fabricate composite materials. Its form may be sheet, tape, or tow. For thermosetting polymer, the polymer has been partially cured to a controlled viscosity called “B stage.” **D3878**

3.1.21 *selvage, n*—the woven edge portion of a fabric parallel to the warp. **D3878**

3.1.22 *tow, n—in fibrous composites*, a continuous, ordered assembly of essentially parallel, collimated continuous filaments, normally without twist. (Synonym – roving) **D3878**

3.1.23 *unidirectional composite, n*—any fiber reinforced composite with all fibers aligned in a single direction. **D3878**

3.1.24 *warp, n*—the yarn running lengthwise in a woven fabric. **D3878**

[4] The last approved version of this historical standard is referenced on www.astm.org.

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

3.1.25 *woven fabric, n*—a fabric structure produced by the interlacing, in a specific weave pattern, of tows or yarns oriented in two or more directions.

3.1.25.1 *Discussion*—There are a large variety of 2D weave styles, e.g., plain, satin, twill, basket, crowfoot, etc.

3.1.26 *yarn, n—in fibrous composites*, a continuous, ordered assembly of essentially parallel, collimated filaments, normally with twist, and of either discontinuous or continuous filaments. Single yarn – an end in which each filament follows the same twist. **D3878**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *1D, 2D, and 3D reinforcement, n*—a description of the orientation and distribution of the reinforcing fibers and yarns in a composite.

3.2.1.1 *Discussion*—In a 1D structure, all of the fibers are oriented in a single longitudinal (x) direction. In a 2D structure, all of the fibers lie in the x-y planes of the plate or bar or in the circumferential shells (axial and circumferential directions) of the rod or tube with no fibers aligned in the z or radial directions. In a 3D structure, the structure has fiber reinforcement in the x-y-z directions in the plate or bar and in the axial, circumferential, and radial directions in a tube or rod.

3.2.2 *axial tensile strength, n*—for a composite tube or solid round rod, the tensile strength along the long axis of the tube or rod. For a composite flat plate or rectangular bar, the tensile strength along the primary structural axis/direction.

3.2.3 *chemical vapor deposition or infiltration, n*—a chemical process in which a solid material is deposited on a substrate or in a porous preform through the decomposition or the reaction of gaseous precursors.

3.2.3.1 *Discussion*—Chemical vapor deposition is commonly done at elevated temperatures in a controlled atmosphere.

3.2.4 *durability, n*—the measure of the ability of a material or structure to endure and maintain its essential and distinctive chemical, physical, mechanical and other functional characteristics in a specific environment of use (temperature, atmosphere, stress, radiation, etc) for a designated period of time.

3.2.5 *fiber interface coating, n—in ceramic composites*, a coating applied to fibers to control the bonding between the fiber and the matrix.

3.2.5.1 *Discussion*—It is common practice in SiC-SiC composites to provide a thin (<3 micrometers) interface coating on the surface of the fibers/filaments to prevent strong bonding between the SiC fibers and the SiC matrix. A weak bond between the fiber and the matrix in the SiC-SiC composite permits the fibers to bridge matrix cracks and promotes mechanical toughness and damage tolerant failure; a strong bond between the matrix and the fiber produces low strain, brittle failure. Fiber interface coatings with controlled composition, thickness, phase content, and morphology/microstructure are used to control that interface strength. (**9, 10**)

3.2.6 *hot press and sinter densification, n—in SiC matrix composites*, a matrix production and densification process in which silicon carbide particulate in the preform are consolidated and sintered together to high density in a die press at high pressures and temperatures.

3.2.6.1 *Discussion*—A sintering additive is often added to the silicon carbide powders to produce liquid phase sintering and accelerate densification.

3.2.7 *infiltration and pyrolysis densification, n—in SiC matrix composites*, a matrix production and densification process in which a liquid silicone-organic polymer precursor is infiltrated/impregnated into the porous perform or the partially porous composite and pyrolyzed to form the silicon carbide matrix.

3.2.7.1 *Discussion*—Pyrolysis of the silicone-organic precursor in an inert atmosphere converts the precursor to a silicon carbide form with the desired purity and crystal structure. The infiltration/pyrolysis process may be iteratively repeated to fill the porosity and build up the density in the composite. (**11**)

3.2.8 *melt infiltration, n—in SiC matrix composites*, the matrix production and densification process in which molten silicon is injected in a preform (containing SiC fibers and SiC and carbon particulate) and the molten silicon reacts with the free carbon to form a bonding silicon carbide matrix. (Synonyms – reaction sintering, liquid silicon infiltration) (**12**)

3.2.9 *primary structural axis, n—in a composite flat plate or rectangular bar*, the directional axis defined by the loading axis/direction with the highest required tensile strength.

3.2.9.1 *Discussion*—The primary structural axis is commonly the axis with the highest fiber loading. This axis may not be parallel with the longest dimension of the plate/bar/structure.

3.2.10 *pyrolysis, n—in SiC matrix composites*, the controlled thermal process in which a silicone-organic precursor is decomposed in an inert atmosphere to form the silicon carbide (SiC) matrix.

3.2.10.1 *Discussion*—Pyrolysis commonly results in weight loss and the release of hydrogen and hydrocarbon vapors.

3.2.11 *rectangular bar, n*—a solid straight rod with a rectangular cross-section, geometrically defined by a width, a thickness, and a long axis length.

3.2.12 *round rod, n*—a solid elongated straight cylinder, geometrically defined by an outer diameter and an axial length.

3.2.13 *round tube, n*—a hollow elongated cylinder, geometrically defined by a outer diameter, an inner diameter, and an axial length.

3.2.14 *silicon carbide – silicon carbide composite, n*—a ceramic matrix composite in which the reinforcing phase consists of continuous silicon carbide filaments in the form of fiber, continuous yarn, or a woven or braided fabric contained within a continuous matrix of silicon carbide. (**13-15**)

3.2.15 *silicon carbide fibers, n*—inorganic fibers with a primary (≥80 weight%) silicon carbide (stoichiometric SiC formula) composition.

3.2.15.1 *Discussion*—Silicon carbide fibers are commonly produced by two methods—the high temperature pyrolysis and sintering of silicone-organic precursor fibers in an inert atmosphere and the chemical vapor deposition of silicon carbide on a substrate filament. (**16**)

3.2.16 *surface seal coatings, n*—an inorganic protective coating applied to the outer surface of a SiC-SiC composite component to protect against high temperature oxidation and/or corrosion attack or to improve wear and abrasion resistance. Such coatings are commonly hard, impermeable ceramic/glass coatings.

4. Significance and Use

4.1 Composite materials consist by definition of a reinforcement phase in a matrix phase. In addition, ceramic matrix composites (CMCs) often contain measurable porosity which interacts with the reinforcement and matrix. And SiC-SiC composites often use a fiber interface coating which has an important mechanical function. The composition and structure of these different constituents in the CMC are commonly tailored for a specific application with detailed performance requirements. The tailoring involves the selection of the reinforcement fibers (composition, properties, morphology, etc), the matrix (composition, properties, and morphology), the composite structure (component fractions, reinforcement architecture, interface coatings, porosity structure, microstructure, etc.), and the fabrication conditions (forming, assembly, forming, densification, finishing, etc.). The final engineering properties (physical, mechanical, thermal, electrical, etc) can be tailored across a broad range with major directional anisotropy in the properties.

4.2 Specifications for specific CMC components covering materials, material processing, and fabrication procedures are developed to provide a basis for fabricating reproducible and reliable structures. Designer/users/producers have to write CMC specifications for specific applications with well-defined composition, structure, properties and processing requirements. But with the extensive breadth of selection in composition, structure, and properties in CMCs, it is virtually impossible to write a "generic" CMC specification applicable to any and all CMC applications that has the same type of structure and details of the commonly-used specifications for metal alloys. This guide is written to assist the designer/user/producer in developing a comprehensive and detailed material specification for a specific CMC application/component with a specific focus on nuclear applications.

4.3 The purpose of this guide is to provide guidance on how to specify the constituents, the structure, the desired engineering properties (physical, chemical, mechanical, durability, etc), methods of testing, manufacturing process requirements, the quality assurance requirements, and traceability for SiC-SiC composites for nuclear reactor applications. The resulting specification may be used for the design, production, evaluation, and qualification of SiC-SiC composites for structures in nuclear reactors.

4.4 The guide is applicable to SiC-SiC composites with flat plate, rectangular bar, round rod, and round tube geometries.

4.5 This guide may also be applicable to the development of specifications for SiC-SiC composites used for other structural applications, discounting the nuclear-specific chemical purity and irradiation behavior requirements.

5. Silicon Carbide-Silicon Carbide Composites for Nuclear Applications

5.1 Silicon carbide-silicon carbide (SiC-SiC) composites are candidate structural materials for use in nuclear reactors, because of their high temperature stability, oxidation resistance, radiation tolerance, and low neutron cross-section compared to metals and for their damage tolerance and tailored anisotropic mechanical and physical properties, compared to monolithic ceramics. **(1-8)**

5.2 SiC-SiC composites are composed of silicon carbide fiber reinforcement in a silicon carbide matrix. The chemical and phase composition, microstructure, and properties of the fibers and the silicon carbide matrix, the fiber architecture (the shape and morphology of the fiber preform, multidimensional fiber distribution, and volume content of the fiber reinforcement), and the composite density and porosity are engineered to give the desired performance properties for the composite. The SiC fibers generally have a fiber interface coating to control the bonding and sliding between the SiC fiber and the SiC matrix. **(13-15)**

5.3 The physical, mechanical, and thermal properties of SiC-SiC composites are determined by the complex interaction of the constituents (fiber, interface coating, matrix, porosity) in terms of the constituent chemistry, phase composition, microstructure, properties, and fractional content; the fiber architecture; the fiber-matrix bonding, and the effect of fabrication on the constituent properties, morphology, and their physical interactions. These factors can be synergistically tailored to produce a structure/component with the desired mechanical, physical, and thermal properties. The SiC-SiC composite properties can be tailored for directional properties by the anisotropic architecture of the silicon carbide fiber reinforcement. **(13-15)**

5.4 Silicon carbide fibers produced by the polymer precursor route are commonly small diameter (5-20 micrometers) continuous filaments. **(16)** The mechanical and thermal properties of the silicon carbide fibers are strongly dependent on the silicon carbide stoichiometry, oxygen and impurity levels, the phase composition and fractions, and the crystallite size and orientation in the fibers. These factors are determined by the precursor chemistry and the fabrication process conditions.

5.5 The silicon carbide fibers are commonly consolidated into high count multifilament tows which can be wound, wrapped or layed-up into 1D structures, woven/layed-up/braided/knitted into 2D structures, or woven/braided/knitted/stitched into 3D structures. Each of these fiber structures are fabricated with defined fiber architectures, offering a wide range of bulk fiber content. Different fiber architectures may have marked reinforcement anisotropy, depending on the relative fiber content in each orthogonal direction.

NOTE 1—Many commercially available SiC-SiC composites consist of stacked fabric plies with a two dimensional woven fabric architecture. The SiC-SiC composite is densified to produce a final structure with orthotropic or quasi-isotropic mechanical and thermal properties.

5.6 The silicon carbide matrix in SiC-SiC composites is commonly produced by four methods: *(1)* a chemical vapor infiltration process, *(2)* an iterative precursor liquid infiltration/

pyrolysis process, *(3)* a silicon melt infiltration process, or *(4)* hot pressing and sintering of SiC powders. The four matrix formation processes use different precursors and different processing conditions, which produce differences in the chemistry, phase composition and fractions, crystallinity, morphology, and microstructure (density, pores, and cracks) in the silicon carbide matrix. Two or more of these matrix densification processes may be combined for a hybrid silicon carbide matrix.

5.7 The interaction of these four variable factor sets [*(1)* silicon carbide fiber type and properties; *(2)* fiber interface coating; *(3)* fiber content, tow structure, and architecture; *(4)* matrix composition and properties, phase content, crystallinity, density, morphology, and porosity] can produce SiC-SiC composites with a wide range of mechanical and physical properties, along with tailored anisotropic properties in the major directions.

NOTE 2—For nuclear applications, SiC-SiC composites made from stoichiometric, high purity, and fully crystalline SiC fibers and matrices are preferred for their physical, chemical, and mechanical property stability in the temperature and high radiation flux conditions of light water fission reactors and high temperature fission reactors. **(1-8)**

6. Product Specifications—Properties, Materials and Processing

6.1 The fibers, matrix, fiber architecture, fiber interface coatings, any surface seal coatings, and the method of manufacture, when combined as a SiC-SiC composite structure, must produce a composite that consistently and reliably meets the performance requirements (chemical, physical, mechanical, and durability) specified by the designer/purchaser/user, applicable codes and standards, and the controlling regulatory agency.

6.2 The engineering properties and characteristics of a composite structure are manufactured into the structure as part of the fabrication process. Specifications for SiC-SiC composites shall be written to define requirements for end product properties (chemical constituents and phase composition, physical properties, mechanical properties, durability, etc.), and manufacturing specifications for starting materials and fabrication. The manufacturing specifications shall include sufficient information to ensure that critical factors and parameters in the starting materials and the manufacturing process are identified and controlled to produce the final structure/component to the defined specification.

6.3 The designer/purchaser/user shall define the specifications for the constituents (chemistry and properties), architecture, final properties, and quality assurance for the SiC-SiC composite.

6.4 The designer/purchaser/user and the manufacturer together shall define the specifications for the materials/processing manufacture and non-destructive testing (NDT) of the SiC-SiC composite.

7. Product Specification—Composite Constituents, Chemical Composition and Purity for Nuclear Applications

7.1 A SiC-SiC composite shall consist of silicon carbide reinforcement fibers in a silicon carbide matrix. The fibers may have a fiber interface coating/treatment to control the bonding between the fiber and the matrix.

7.2 The composite may have a surface coating to seal the composite against gas and liquid penetration/escape and to protect the composite from oxidation or environmental degradation.

7.3 The designer/purchaser/user shall specify the required composite constituents and structures in terms of silicon carbide fibers, fiber interface coatings, silicon carbide matrix, and surface seal coatings. The specification shall list sources, chemical compositions and phase content, component fractions and morphology, reinforcement architecture, and seal coating requirements, as required by the designer/purchaser/user. Section 11 describes the manufacturing process specification requirements for fibers, interface coatings, matrix, architecture, and seal coatings.

7.4 For nuclear applications impurity levels in SiC-SiC composites (and any surface seal coatings) have to be carefully controlled to minimize parasitic neutron absorption, oxidation promoting catalysis, nuclear activation impurities, corrosion promotion impurities, and fissionable elements. The designer/purchaser/user shall specify the requirements and test methods for chemical purity based on the defined requirements for the specific nuclear application. (An example of chemical purity requirements for nuclear grade graphite (from D7219) is given in Table X1.1.)

NOTE 3—Table X1.2 (from D7219) contains a list of chemical impurities typically found in nuclear grade graphite and carbon. The impurities are categorized as neutron absorbing impurities, oxidation promoting catalysts, activation relevant impurities, metallic corrosion relevant impurities, and fissile/fissionable elements. The suggested limits represent the reactor designer's preferences for chemical purity in graphite, which may be extended to silicon carbide.

7.5 The designer/purchaser/user shall specify boron equivalent limits and test methods for the specific nuclear application. The boron equivalent shall be calculated in accordance with Practice C1233 as specified for nuclear grade graphite (referenced in Table X1.1).

7.6 Each SiC-SiC composite production lot sampled in accordance with Section 14 shall conform to the requirements for chemical purity and boron equivalency specified by the designer/ purchaser/user.

8. Product Specification—Physical Properties

8.1 The designer/purchaser/user shall specify the required minimum/maximum values for the specified physical, thermal, and electrical properties of SiC-SiC composites based on the desired performance properties; the component constituents, fractions, and properties; the reinforcement architecture; and the final porosity fraction.

8.2 The physical, thermal, and electrical properties, of SiC-SiC composites that are of primary and secondary interest are listed in Table 1 with the recommended ASTM test standards. The selection of specific physical, thermal, and electrical properties for the specification will depend on the design requirements for the CMC component. Other properties (not included in this list) may be specified by the designer/purchaser/user, based on application-specific requirements.

TABLE 1 Physical, Thermal and Electrical Properties of SiC-SiC Composites

NOTE 1—For round rods and tubes, anisotropy should be defined in terms of axial, radial, and tangential (hoop) directions, not x, y, and z.

NOTE 2—Thermal expansion, thermal conductivity, electrical resistivity, and emissivity data may be anisotropic depending on fiber architecture and should be measured in the major directions.

NOTE 3—Physical properties may be strongly dependent on bulk porosity content and on localized porosity concentrations (which may be inhomogeneously distributed).

	Units	ASTM Test	Priority	Anisotropy
Bulk Density by Physical Measurement	g/cm^3	C559, C838	Primary	No
Apparent Porosity and Bulk Density by Immersion	% and g/cm^3	C1039	Primary	No
Constituent (Fiber, Matrix) Bulk Fraction	%	D3171 (Method 2)	Primary	No
Fiber Fraction–Directional	%	By calculation	Primary	Yes (x,y,z)
Matrix SiC Crystallinity (Phase Content and Fractions)	%	TBD	Primary	No
Fiber SiC Crystallinity (Phase Content and Fractions)	%	TBD	Primary	No
Linear Thermal Expansion	ppm/°C	C1470, E228, E289	Secondary	Yes (x,y,z)
Thermal Conductivity – (Diffusivity)	W/(m-K) – (m^2/s)	C1470, C714, E1461	Secondary	Yes (x,y,z)
Specific Heat	J/(g-K)	C1470, D2766, E1269	Secondary	No
Emittance, Emissivity	nd	C1470, E408, E423	Secondary	Yes (x,y,z)
Electrical Resistivity	Ohm-m	C611[A]	Secondary	Yes (x,y,z)
Porosity Content and Structure (Mercury Porosimetry)	TBD	D4284	Secondary	No
Surface Area (BET)	m^2/g	C1274	Secondary	No
Permeability	L /(m^2-s)	C577[A]	Secondary	Yes (x,y,z)
Surface Roughness	TBD	Surface Profilometry ASME B46.1	Secondary	Yes (x,y,z)

[A] Modification of this test method may be required for SiC-SiC composites.
nd = no dimensions.
TBD = to be determined.

8.2.1 The designer/purchaser/user may define anisotropy requirements and limits for designated physical properties.

8.3 The designer/purchaser/user may specify requirements for descriptive statistics and limits (test count, mean, standard deviations, coefficient of variation, minimum/maximum values, etc) for the designated physical properties (see E2586).

8.4 *Elevated Temperatures*—The designer/purchaser/user may also specify requirements for thermal and electrical properties at specified elevated temperatures, determined by the performance requirements.

8.5 The designer/purchaser/user may specify requirements for anisotropy in thermal and electrical properties, determined by the performance requirements.

8.6 Variability in physical properties (in-piece anisotropy, in-piece volumetric, piece-to-piece, and lot-to-lot) may be of direct interest to the manufacturer and the designer/purchaser/user.

8.7 Other physical properties may be specified by the designer/purchaser/user [see Composite Materials Handbook CMH-17, Volume 5 (CMC Handbook)].

8.8 Each SiC-SiC composite production lot shall be sampled in accordance with Section 14.

9. Product Specification—Mechanical Properties

9.1 The designer/purchaser/user shall specify the required maximum/minimum values for the selected mechanical properties of SiC-SiC composites considering anisotropy and based on the desired performance properties, the component constituents and fractions, and the reinforcement architecture.

9.2 Mechanical property specifications for each kind of stress test should include (per designer/ purchaser/user requirements): *(1)* the ultimate strength and strain, *(2)* the fracture strength and strain, *(3)* the proportional limit stress and strain, *(4)* elastic modulus, and *(5)* representative stress-strain curves.

9.3 The stress-strain response of a SiC-SiC composite can vary widely, ranging from linear elastic brittle failure to very high strain failure with major damage accumulation and pseudo-ductility (see Fig. 1). The stress-strain response of the composite depends on the interaction of many factors—fiber properties, architecture, and volume fraction, matrix density and properties, matrix-fiber bonding, stress alignment with the reinforcement axes, and deformation/damage mechanisms.

9.4 The mechanical properties of SiC-SiC composites that are of interest are listed in Table 2 with the recommended ASTM test standards. The selection of specific properties for the specification will depend on the design requirements for the specific SiC-SiC component.

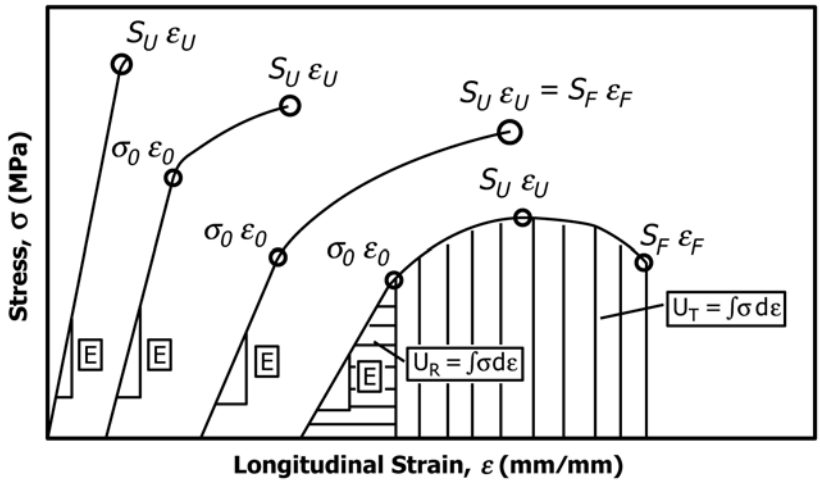
9.4.1 *Elevated Temperatures*—The designer/purchaser/user may specify requirements for mechanical properties at specific elevated temperatures, determined by the performance requirements.

9.4.2 *Anisotropy*—The designer/purchaser/user may define anisotropy requirements and limits for designated mechanical properties.

9.4.3 The designer/purchaser/user may specify requirements for descriptive statistics and limits (test count, mean, standard deviations, coefficient of variation, minimum/maximum values, etc) for the designated mechanical properties (see E2586).

9.4.4 The designer/purchaser/user may specify requirements for Weibull modulus and Weibull characteristic strength for selected mechanical properties (see C1239 and C1683).

9.5 Variability in mechanical properties (in-piece volumetric, in-piece anisotropy, piece-to-piece, and lot-to-lot) may be of direct interest to the manufacturer and the designer/



S_U = ultimate strength, MPa
ϵ_U = ultimate strain, %
S_F = fracture strength, MPa
ϵ_F = fracture strain, %
σ_O = proportional limit stress, MPa
ϵ_O = proportional limit strain, %
E = elastic modulus, GPa
U_R = modulus of resilience (J/m^3) integral of σ from 0 to ϵ_O strain
U_T = modulus of toughness (J/m^3) integral of σ from 0 to ϵ_F strain

FIG. 1 Examples of Different SiC-SiC Stress-Strain Curves

TABLE 2 Mechanical Properties of SiC-SiC Composites

NOTE 1—Mechanical properties may be strongly anisotropic (axial, transverse, off-axis, etc) depending on fiber architecture and directional fiber fraction and should be measured in the major directions.

NOTE 2—Mechanical properties may be strongly dependent on bulk porosity content and on porosity concentrations (which may be inhomogeneously distributed).

	Units	ASTM Test – Flats-Bars	ASTM Test – Rods/Tubes
Tensile Properties (ultimate, fracture, PropL)	MPa & strain	C1275, C1359	C1773
Flexure Properties (ultimate, fracture, PropL)	MPa & strain	C1341	[B]
Compression Properties (ultimate, fracture, PropL)	MPa & strain	C1358	[B]
Shear Properties (ultimate, fracture, PropL)	MPa & strain	C1292, C1425	[B]
Transthickness Tensile Properties (ultimate, fracture, PropL)	MPa & strain	C1468	[B]
Hoop Strength Properties (ultimate, fracture, PropL)	MPa & strain	NA	[B]
Elastic/Shear Modulus by Mechanical Loading	GPa	E111, E143	E111[A] , E143[A]
Elastic/Shear Modulus by Sonic Resonance	GPa	C1198	C1198[A]
Elastic/Shear Modulus by Impulse Excitation	GPa	C1259	C1259[A]
Elastic Modulus by Sonic Velocity	GPa	C769	C769[A]
Poisson's Ratio	nd	E132	E132
Modulus of Resilience (in Tension)	J/m^3	C1275, C1359	C1773
Modulus of Toughness (in Tension)	J/m^3	C1275, C1359	C1773
Open Hole Tensile Strength Properties	MPa & strain	D5766[A]	[B]
Open Hole Compression Strength Properties	MPa & strain	D6484[A]	[B]
Notch Tensile Strength Properties	MPa & strain	[B]	[B]
Notch Compression Strength Properties	MPa & strain	[B]	[B]
Pin Bearing Strength Properties	MPa & strain	D5961[A]	[B]
Crack Growth Resistance/ Strain Energy Release Rate/ (Fracture Toughness) (Interlaminar and Translaminar)	kJ/m^2	D5528[A] , D6671[A] , E1922[A]	[B]

[A] Modification of this polymer matrix composite test method may be required.
[B] New test methods are required.
nd = no dimensions.

purchaser/ user. The designer/purchaser/user may specify statistically-based requirements to characterize variability across the different factors.

9.6 Other mechanical property requirements may be specified by the designer/purchaser/user [see Composite Materials Handbook CMH-17, Volume 5 (CMC Handbook)].

9.7 Each SiC-SiC composite production lot shall be sampled in accordance with Section 14.

10. Product Specification—Durability Properties

10.1 The durability of SiC-SiC composites over time under reactor environment conditions is a principal engineering design concern. In a nuclear reactor, the composites must maintain a defined set of chemical, physical, and mechanical properties for extended periods of time under defined conditions of fast neutron radiation exposure, static and cyclic stress at elevated temperatures, and high temperature oxidation/corrosion (O_2/H_2O/CO_2/N_2, et al.) exposure. This requires that physical changes in the composite structure (chemistry, phases, crystallinity, grain size, mass loss/gain, porosity, corrosion products, flaw populations) and degradation of physical and mechanical properties all be understood and controlled in terms of the fiber, the interface coatings, the matrix, porosity, and surface seal coatings. Any changes in structure and properties will depend on the combined effects of time, radiation levels, temperature, stress, and oxygen/corrosion concentration. **(2, 4, 8, 17-22)**

NOTE 4—Under neutron radiation at reactor operating temperatures, the composite should undergo minimal swelling, shrinkage, dimensional changes, phase changes, pore formation, and microcracking that may degrade the physical and mechanical properties and the functionality of the composite.

NOTE 5—Different silicon carbide fibers and silicon carbide matrices will have different susceptibility to radiation damage based on crystal structure and impurities. The radiation damage effects should be assessed and understood for each specific component, including matrix, fiber, interface coating, and surface seal coating.

10.2 Oxidation/corrosion effects at reactor operating temperatures must by controlled and managed in terms of chemical reactions, phase changes, mass loss/gain, dimensional changes, and corrosion products. Any degradation of physical and mechanical properties of the composite as a whole, must be managed and minimized, including any fiber interface coating and surface seal coating effects.

10.3 Stress effects must be understood and controlled in terms of crack growth, flaw initiation, fatigue degradation, creep strain, and stress-rupture, all as a function of temperature, time, stress levels, and oxidation/corrosion conditions.

10.4 The designer/purchaser/user shall specify the durability requirements (physical, mechanical, etc.) for the SiC-SiC composites under defined conditions of time, temperature, neutron irradiation, stress, oxidation conditions, and corrosion concentrations. Durability requirements are commonly defined as a "not-to-exceed" maximum % change in designated properties as a function of exposure conditions and time. The specification shall define the experimental test methods and the required exposure-conditioning parameters for determining the physical, chemical, and mechanical durability. (Table 3 is a list of durability factors that need to be considered and possibly specified, depending on performance requirements.)

10.5 Other durability requirements may be specified by the designer/purchaser/user [see Composite Materials Handbook CMH-17, Volume 5 (CMC Handbook)].

10.6 Each SiC-SiC composite production lot shall be sampled and tested per the designer/purchaser/user requirements for durability testing.

11. Manufacturing Process Specifications

11.1 The fibers, matrix, fiber architecture, fiber surface treatments, any component coatings, and the method of manufacture, when combined as a composite structure, must produce a composite component that consistently and reliably meets the performance (chemical, physical, mechanical, and durability) requirements specified by the designer/purchaser/user, applicable codes and standards, and the controlling regulatory agency.

11.2 The chemical, physical, mechanical, and durability properties of a composite structure are manufactured into the structure as part of the manufacturing process. Therefore, material and process specifications used to produce composite structures need to contain sufficient information to ensure that critical parameters in the starting materials and the manufacturing process are identified and controlled to produce the final structure to the defined specification.

11.3 The designer/purchaser/user and the manufacturer shall define the manufacturing specifications used for the SiC-SiC composite production and qualification. Typical material and process specifications should contain the following information, as a minimum:

TABLE 3 Durability Testing of SiC-SiC Composites

NOTE 1—Tested against the specified range of temperature, time, irradiation, stress, oxidation/ corrosion, and performance conditions.

	ASTM Test
Neutron Irradiation Exposure Testing– Dimensional Stability, Swelling, Radiation Creep, and Changes in Physical and Mechanical Properties	E261, C625[A,B]
Oxidative Mass Loss in Air and Reactor Environment (with and without surface seal coat)	C1179, D7542[A]
Oxidation Exposure Testing (Changes in Physical and Mechanical Properties)	[B]
Corrosion Exposure Testing (Changes in Physical and Mechanical Properties)	[B]
Creep Rates and Creep Rupture (Tensile, Flexure, and Compression) (Air and/or Inert Conditions)	C1291[A], C1337
Fatigue (Tensile, Flexure, and Compression) (Air and Inert Conditions)	C1360[A]
Slow Crack Growth (Tensile, Flexure, and Compression) (Air and/or Inert Conditions)	[B]
Thermal Shock Resistance	C1525[A]
Impact Damage (Tensile, Flexure, and Compression)	D7136[A], D7137[A]
Wear, Abrasion, and Erosion Resistance	[B]
Thermomechanical Cyclic Effects	[B]

[A] Modification of this test method may be required.
[B] New additional test methods are required.

11.3.1 *Raw Material Constituent Specifications:*

11.3.1.1 *Silicon Carbide Fiber/Tow*—The specification shall define the source, method of manufacture, manufacturer's specification ID, oxygen content, impurity levels, crystal structure, grain/crystallite size, filament diameter, density, filaments count and tow architecture, mechanical properties, and thermal properties along with defined tolerances. (See C1557, D3800, and D4018.)

11.3.1.2 *Fiber Surface Treatment and Interface Coatings*—If the raw material silicon carbide fiber has a surface treatment or an interface coating, the specification shall define the manufacturing source, manufacturer's specification ID, chemical and phase composition, impurity levels, density, porosity, thickness, crystallinity, grain/crystallite size, method of manufacture, and any other required properties, along with defined tolerances.

11.3.1.3 *Matrix Precursors and Constituents*—The specification shall define the manufacturer's specification, chemical composition, impurity limits, and any other required (physical, thermal, chemical, processing etc) properties along with tolerances.

11.3.1.4 *Precursors and Constituents for Protective Seal Coatings*—The specification shall define chemical composition, impurity limits, manufacturer's specification and any other required properties, along with tolerances.

11.3.2 *Precursor and Constituent Materials Acceptance*—The composite manufacturer shall establish a process control document (PCD) that defines and documents the quality control criteria and testing methods for all incoming raw materials.

11.4 *Fiber Reinforcement Architecture and Fabrication*—The fiber reinforcement in SiC-SiC composites is produced by the consolidation and densification of 1D. 2D, and 3D fiber preforms into the finished composite. The specific architecture (1D, 2D, 3D) is defined to give the desired bulk and directional properties in the finished composite. The preform is infiltrated with a liquid or gas precursor at various stages of manufacture to produce the desired silicon carbide matrix.

11.4.1 Specifications for the fiber architecture define the overall fiber content (by weight or volume) and the details of the fiber architecture, in terms of the fiber content and morphology (winding, weave, braid, knit, etc.) in the 1D, 2D, and 3D formats along defined axes, as needed.

11.4.2 For polymer precursors, the fiber in 1D or 2D forms may be preinfiltrated with partially cured polymer to form a "Prepreg." The "prepreg plies" are stacked and consolidated under pressure and heat to form an intermediate stage composite.

11.4.3 *Fiber Architecture Specification*—The fiber architecture can be described as one- dimensional (1D), two-dimensional (2D), and three dimensional (3D) structures. Each structure type has specific descriptive elements.

11.4.3.1 *Tow/Yarn (1D)*—The tow filament count, linear tow density, and tow twist amount and direction.

(1) Infiltrated/prepregged Tow—The resin type, resin weight fraction, and the resin B-stage condition in the tow.

(2) Winding Description—The fabric areal weight, yarn volume percentage, and winding process description.

11.4.3.2 *Fabric Lay-up (2D) Specifications*—2D composites are formed by the stacking and consolidation of 2D fabrics or 1D tapes.

(1) 2D Woven Fabric—Fabric weave style/description, fabric nominal areal weight, end/warp count, pick/fill count, linear density (warp and fill), weave/unit cell size.

(2) Uniaxial (1D) Tape—Nominal areal weight of the tape, end count.

(3) 2D braided Fabric—Braid description, filament counts (axial and braid yarns), braid angle, yarn percentage (axial and braid yarns), axial yarn spacing in braids.

(4) Prepregged Fabric—The resin type, resin weight fraction, and the resin B-stage condition in the prepreg.

11.4.3.3 *2D Lay-Up*—Ply count, per-ply thickness, lay-up stacking/orientation description (see D6507).

11.4.3.4 *3D Preform Specification*—3D fabric performs can be defined as three structures: 2D fabrics that are stitched together through the thickness, 3D woven preforms, and 3D braided preforms.

(1) Stitched/Needled Fabric—The fabric description and layup description (see 11.4.3.2). Description of the stitching yarn (in terms of the filament count, linear density, twist pitch, and end count) and the stitching density and pattern.

(2) 3D Woven Preform—Weave and interlock description, filament counts (warp, weft, angle, weaver yarns), end counts and yarn percentages (warp, weft, angle, weaver yarns), angle of angle yarn.

(3) 3D Braid Preform—Braid description, filament counts (axial and braid yarns), braid angle, yarn percentages (axial and braid yarns), axial yarn spacing in braids.

11.4.4 *Fiber Reinforcement Architecture Process Control*—The reinforcement preform/architecture manufacturer should establish a process control document (PCD) that defines and documents the key aspects of the reinforcement architecture production process, lists all raw material constituents, defines key process parameters, and establishes statistical process control (SPC) procedures and quality control standards.

11.5 *Composite Fabrication Process Control*—The composite manufacturer should establish a process control document (PCD) that defines and documents the key aspects of the composite production process, lists all raw material ingredients, defines key process parameters, and establishes statistical process control (SPC) procedures and quality control standards for composite. This PCD should cover all the critical processing steps for the SiC-SiC composites, to include (but not limited to):

11.5.1 *Personnel Qualification.*

11.5.2 *Equipment and Tooling Description.*

11.5.3 *Facility Description.*

11.5.4 *Input Materials* (see section 11.3) and storage and handling methods.

11.5.5 *Fabrication Process Instructions,* to include:

11.5.5.1 Fiber preform fabrication and consolidation (see section 11.4).

11.5.5.2 Matrix infiltration and densification—methods, process temperatures, times, and conditions. Depending on the method of composite and matrix formation, this will cover filament winding, polymer infiltration and curing, prepreg

lamination, vapor infiltration/ deposition, pyrolysis, tooling and fixturing, reinfiltration, matrix densification, silicon melt infiltration, heat treatment and reaction temperatures, times and atmospheres, etc.

11.5.5.3 Sintering of the silicon carbide composite—sintering additives, temperatures, pressure conditions, times, atmosphere, process conditions, and final density of the silicon carbide and porosity content of the matrix.

11.5.5.4 Final matrix condition—chemical composition, phase composition and fractions, crystal structure, grain/crystallite size, porosity structure and fraction, and microstructure description.

11.5.5.5 Finish machining of the composite—machining methods (cutting, drilling, turning, milling, grinding, polishing, electro-discharge, laser, water-jet, etc), tools, and parameters.

11.5.5.6 Structural component seal coatings—chemical and phase composition, method of fabrication, final thickness, morphology and grain structures, porosity, permeability, hardness, and adhesion.

11.5.6 *Structural Component Acceptance and Quality Control*—See Sections 14 and 15.

12. Other Requirements

12.1 *Storage and Handling during Fabrication*—The raw materials, intermediate stage structures, and the finished composites shall be protected from damage and contamination that would degrade the chemical, physical, and mechanical properties of the composites.

12.2 *Machining*—Finished composite components may be machined to finished dimensions and tolerances and also machined for detail features (holes, slots, notches, etc) as specified by the designer/purchaser/user. A suitable machining procedure shall be determined and defined by the manufacturer and the designer/purchaser/user to minimize loss of structural integrity and mechanical properties.

12.3 *Seal Coating*—If required by the designer/purchaser/user, a seal coating shall be fabricated on the finished structure after machining. The seal coating shall not reduce the mechanical function or the durability of the composite structure in the reactor environment. The chemical and phase composition, morphology, production method, and performance of the coating shall be determined and specified by the designer/purchaser/user.

12.4 *Identification and Traceability*—Each composite component shall be marked with a unique identification number, so that it is traceable for constituents, fabrication source and lot, critical fabrication conditions, and as-manufactured properties. The method of marking shall not mechanically damage the surface of the component.

13. Quality System

13.1 The quality system for SiC-SiC composites should include procedures that ensure the quality of incoming materials, the control of in-process manufacturing methods, and testing performed to evaluate the end product for conformity to design requirements. The quality system should include standards to be used for nondestructive and destructive tests, visual inspection techniques during the manufacturing process, and product final acceptance. The standards that determine the acceptance or rejection of manufacturing-induced defects and damage should take into account the process and the inspection capability.

14. Composite Component Lot Qualification and Testing

14.1 The specification shall include a qualification and testing plan that includes:

14.2 A statistical sampling plan shall be developed by the manufacturer and the designer/producer/user to support the testing plan. The plan shall describe the number of composite components and witness sections to be sampled and tested for product property acceptance for each production lot.

14.3 *Chemical Properties*—A representative number of composite components shall be sampled for chemical impurities, per Section 7. The number and identity of the composite components selected from each lot shall be in accordance with the statistical plan agreed with the designer/purchaser/user.

14.4 *Mechanical, Physical and Durability Properties*—A cutting plan shall be agreed between the designer/purchaser/user and manufacturer. The cutting plan shall describe the location, number and orientation of the test specimens required for property determinations as selected from Sections 8, 9 and 10 of this specification. The cutting plan shall consider property gradients and anisotropy introduced by forming and processing. The number of each type of specimen defined by the cutting plan shall be sufficient to yield statistically significant data.

14.5 The qualification and testing section of the specification may include any other appropriate test methods and requirements, such as proof testing, as defined by the designer/producer/user.

15. Final Inspection and Non-Destructive Testing (NDT)

15.1 SiC-SiC composite components shall be visually inspected for surface flaws and defects. The type, maximum size, location and allowable number of disqualifying surface flaws shall be defined by the designer/purchaser/user and the manufacturer and described in the purchase specification.

15.2 A statistical sampling and NDT plan shall be developed by the manufacturer and agreed with the designer/purchaser/user. The plan shall describe the number of composite components and witness sections to be sampled and non-destructively tested (NDT) to screen for internal defects. The appropriate NDT method/s (radiography, tomography, ultrasonics, thermography, etc.) and the type, location, maximum size and number of internal defect/s acceptable shall be defined in agreement between the designer/purchaser/user and the manufacturer and be described in the purchase specification.

16. Rejection

16.1 SiC-SiC composite components failing requirement specifications for chemical purity, physical properties, mechanical properties, durability, or non-destructive testing shall be rejected.

17. Certification and Quality Assurance

17.1 The manufacturer shall certify that the SiC-SiC composite meets the requirements of the purchase specification and all regulatory codes and specifications, as identified in the designer/purchaser/user's specification.

17.2 The manufacturer of nuclear grade SiC-SiC composites furnished per the purchase specification shall comply with the applicable quality assurance requirements of ASME NQA-1 as identified by the designer/purchaser/user's specification.

18. Product Marking

18.1 Each composite component shall be marked with a permanent unique number, which shall be traceable to component pedigree and process history as specified in section 12.4. The method of marking shall not mechanically damage the surface of the component.

19. Packaging, Package Marking, and Storage

19.1 Packaging of the finished composite component shall be as per the designer/purchaser/user's specification to protect the composite component during handling, transportation, and storage.

19.2 Storage of the finished composite components prior to shipping shall be in a manner such that no damage or degradation is incurred.

20. Keywords

20.1 chemical properties; durability; fabrication; mechanical properties; nuclear reactor; physical properties; processing; silicon carbide composites; silicon carbon fiber; specification; thermal properties

APPENDIX

(Nonmandatory Information)

X1. NUCLEAR GRAPHITE IMPURITY TABLES (FROM ASTM D7219)

X1.1 See Tables X1.1 and X1.2.

TABLE X1.1 Chemical Purity Requirements for Carbon-Carbon Composites in Nuclear Applications (derived from D7219)

Test	ASTM Test	Low Purity (ppm)	High Purity (ppm)
Ash Content	C781	1000 maximum	300 maximum
Chemical Impurity – Ca	D5600	<100	< 30
Chemical Impurity – Co	D5600	<0.3	< 0.1
Chemical Impurity – Fe	D5600	<100	< 30
Chemical Impurity – Cs	D5600	<0.3	< 0.1
Chemical Impurity – V	D5600	<250	< 50
Chemical Impurity – Ti	D5600	<150	< 50
Chemical Impurity – Li	D5600	<0.6	< 0.2
Chemical Impurity – Sc	D5600	<0.3	< 0.1
Chemical Impurity – Ta	D5600	<0.3	< 0.1
Boron Equivalent	C1233	10 maximum	2 maximum
Chemical Impurity – S	C816	To be determined	To be determined

TABLE X1.2 Graphite Impurities List and Limits for Nuclear Graphites[A]

Impurity Category	Element	Symbol	Suggested Limits, ppm	Remark
OPC	Aluminum	Al	< 10	Suggested based on typical maximum observed[B] values and possible contribution to oxidation
OPC	Barium	Ba	< 10	Suggested based on maximum observed values and possible contribution to oxidation
NAI	Boron	B	< 1.0	Strong neutron absorber. Difficult to remove from graphite Suggested value is well above maximum observed values for purified grades
OPC	Calcium	Ca	< 10	Suggested based on maximum observed values and contribution to catalytic oxidation
ARI/NAI	Cadmium	Cd	< 0.05	Suggested based on maximum observed values and contribution to neutron absorption and activity
ARI	Cesium	Cs	< 0.05	Suggested. Not routinely analyzed
ARI/MCRI	Chlorine	Cl	< 5	Suggested based on maximum observed value for electrographite in fuel matrix graphite Not routinely analyzed – analysis may be problematic and prone to scatter. Active isotope is ^{36}Cl (gamma emitter with extremely long half-life)
OPC	Copper	Cu	< 10	Based on maximum observed values
ARI	Cobalt	Co	< 0.05	Suggested based on maximum observed values and contribution to neutron absorption. Problem isotope is ^{60}Co (gamma emitter with relatively long half-life)
NAI	Dysprosium	Dy	< 0.05	Suggested value. Element is not routinely analyzed Absorbency greater than natural boron but EBC factor relatively low
ARI/NAI	Europium	Eu	< 0.05	Suggested based on maximum observed values and contribution to neutron absorption/activity
NAI	Gadolinium	Gd	< 0.05	Suggested based on maximum observed values and contribution to neutron absorption
OPC	Iron	Fe	< 10	Suggested based on maximum observed values and contribution to catalytic oxidation
OPC	Lead	Pb	< 10	Suggested based on maximum observed values and contribution to catalytic oxidation
ARI	Lithium	Li	< 0.05	Suggested based on maximum observed values and contribution to ^{3}H (tritium) formation
OPC	Magnesium	Mg	< 10	Suggested based on maximum observed values
OPC	Manganese	Mn	< 10	Suggested based on maximum observed values
MCRI/NAI	Mercury	Hg	< 1.00	Suggested based on maximum observed values and contribution to neutron absorption and potential for metallic corrosion
OPC	Nickel	Ni	< 5	1. Suggested based on maximum observed values 2. Possible contribution to ^{60}Co
ARI	Nitrogen	N	to be determined[C]	Not routinely analyzed – analysis is problematic and limited to surface. Source of ^{14}C (beta emitter with long half-life that may enter coolant gas)
OPC	Potassium	K	< 5	Based on graphite vendor proposed value
NAI	Samarium	Sm	< 0.05	Suggested based on maximum observed values and contribution to neutron absorption
ARI	Scandium	Sc	< 0.05	Suggested based on maximum observed values and contribution to activity
OPC	Silicon	Si	< 10	Suggested based on maximum observed values
OPC/ARI	Silver	Ag	< 10	Suggested based on maximum observed values
OPC	Sodium	Na	< 5	Based on graphite vendor proposed value
OPC/ARI/MCRI	Sulfur	S	< 5.0	Suggested based on maximum observed value for electrographite in fuel matrix graphite. Not routinely analyzed. Significant for metallic corrosion, oxidation and activation
ARI	Tantalum	Ta	< 0.05	Suggested based on maximum observed values and contribution to activity
FFE	Thorium	Th	< 2.00	Based on graphite vendor proposed value
OPC	Titanium	Ti	< 1.00	Suggested based on maximum observed values and contribution to catalytic oxidation
NAI	Tungsten	W	< 1.00	Suggested based on maximum observed values and contribution to neutron absorption
FFE	Uranium	U	< 0.05	Based on graphite vendor proposed value
OPC	Vanadium	V	< 1.00	Suggested based on maximum observed values and contribution to catalytic oxidation

[A] The suggested limits are regarded as being relevant to the high purity grades in this specification.
[B] The estimated limits on most of the elements have been based on typical maximum observed values for purified pebble bed modular reactor reflector grades. A conservative margin has been applied to these values.
[C] Data are not currently available to establish this value.
Key:
ARI = activation relevant impurities.
FFE = fissile/fissionable element.
MCRI = metallic corrosion relevant impurities.
NAI = neutron absorbing impurities.
OPC = oxidation promoting catalysts.

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