Composites: Part A 30 (1999) 561–567

composites
Part A: applied science and manufacturing

Development of test standards for continuous fiber ceramic composites in the United States

Edgar Lara-Curzio[a,*], Michael G. Jenkins[b]

[a]Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6069, USA
[b]Department of Mechanical Engineering, University of Washington, Seattle, WA 98195-2600, USA

Abstract

Standardization activities in the United States for continuous fiber-reinforced ceramic composites (CFCCs) are reviewed. This brief review focuses on the development of test standards by subcommittee C28.07 of the American Society for Testing and Materials (ASTM) on the drafting of a section of a design code for ceramic and ceramic matrix composite components as part of the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code, and on the development of a set of volumes on ceramic matrix composites for Military Handbook 17 on composites. The participation of the US in the international harmonization of standards for CFCCs is also reviewed. Published by Elsevier Science Limited.

Keywords: CFCCs; International harmonization; Test standards

1. Introduction

Continuous fiber-reinforced ceramic composites (CFCCs) have been the focus of intensive developmental efforts over the last 15 years. These efforts have been driven to a large extent by the promise of substantial economic and environmental benefits if CFCCs are used in military and energy-related technologies, particularly at elevated temperatures [1]. Because the commercial diffusion and industrial acceptance of CFCCs can be hampered by lack of standard test methods, databases or design codes, initial standardization efforts concurrent with the development of these materials have focused on methods for the mechanical evaluation of test specimens, and on the drafting of design codes.

2. Test standards

In the US, the American Society for Testing and Materials (ASTM) has spearheaded the widespread introduction of standard test methods for advanced ceramics and ceramic composites. In 10 years since its inception, ASTM committee C28 on Advanced Ceramics has been responsible for the creation of over 25 standards. These standards range from clones of prior ASTM standards (with some new provisions) to complex, innovative documents tailored specifically to advanced ceramics [2]. Committee C28 of the ASTM is structured into eight subcommittees, as indicated in Fig. 1. A workshop organized by the National Institute for Standards and Technology (NIST) in February 1990 helped set the stage for the establishment of subcommittee C28.07 on ceramic matrix composites [3]. Since its establishment in 1991, subcommittee C28.07 has been responsible for formalizing seven full consensus standard test methods for CFCCs and for drafting several other documents that are currently undergoing the ASTM's internal balloting process. Table 1 lists the existing standards developed for CFCCs by subcommittee C28.07. In addition to its work of developing standards, subcommittee C28.07 has been responsible for informing the composites community of the progress in the standardization process through the organization of workshops and symposia.[1]

The work of subcommittee C28.07 has been organized around task groups, which are responsible for the preparation of drafts, and for the progress of these documents through the balloting and approval process at the subcommittee, committee and society levels. Table 2 lists the task groups currently existing in C28.07.

Other efforts for standardization of test methods for

* Corresponding author.

[1] ''Workshop on Thermal and Mechanical Test Methods and Behavior of CFCCs'', Montreal, Canada, June 1994; ''Symposium on Thermal and Mechanical Test Methods and Behavior of CFCCs'', Cocoa Beach, FL, 8–9 January 1996; ''Workshop on Thermomechanical Tests for CFCCs'', Bozeman, MT, 18 October 1994.

1359-835X/99/$ - see front matter Published by Elsevier Science Limited.
PII: S1359-835X(98)00150-X

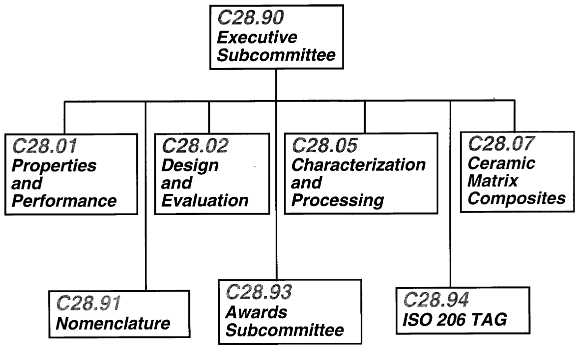

Fig. 1. Structure of ASTM committee C28 on advanced ceramics.

CFCCs in the US include the development of non-consensus, industry-accepted standards developed by the NASA/General Electric/Pratt and Whitney Enabling Propulsion Materials (EPM) Program. These documents, which predate ASTM standards, are listed in Table 3.

In the following sections, the more relevant features of existing ASTM standard test methods for CFCCs are reviewed.

2.1. Tensile testing

A survey of US CFCC manufacturers, conducted in 1991, provided insights into the needs and priorities of this industry for test standards [4]. To address those needs and priorities, the first standard developed for CFCCs by ASTM subcommittee C28.07 was test method C1275 for Monotonic Tensile Strength of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross-Section Specimens at Ambient Temperatures. Later, this standard test method became a template for other documents involving tensile testing, such as C1337 (Standard Test Method for Creep and Creep Rupture of Continuous Fiber-Reinforced Advanced Ceramics under Tensile Loading), C1359 (Standard Test Method for Monotonic Tensile Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross-Sections at Elevated Temperatures) and C1360 (Standard Practice for Constant-Amplitude, Axial, Tension–Tension Cyclic Fatigue of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperatures).

The most important issues addressed by test method C1275 are gripping devices, test system alignment, strain measurements, specimen geometries, specimen preparation, mode and rate of testing, and data acquisition. Although C1275 allows the use of any specimen geometry if it meets the gripping, fracture location and fracture requirements prescribed in the document, in the case of

Table 1
Existing standards under the jurisdiction of ASTM subcommittee C28.07 on ceramic matrix composites

C1275-95	Standard test method for monotonic tensile strength testing of continuous fiber-reinforced advanced ceramics with solid rectangular cross sections at ambient temperatures
C1292-95	Standard test method for shear strength of continuous fiber-reinforced advanced ceramics at ambient temperatures
C1337-96	Standard test method for creep and creep rupture of continuous fiber-reinforced advanced ceramics under tensile loading
C1341-96	Standard test method for flexural properties of continuous fiber reinforced advanced ceramics
C1358-97	Standard test method for monotonic compressive strength testing of continuous fiber-reinforced advanced ceramics with solid rectangular cross-sections at ambient temperatures
C1359-97	Standard test method for monotonic tensile strength testing of continuous fiber-reinforced advanced ceramics with solid rectangular cross-sections at elevated temperatures
C1360-97	Standard practice for constant-amplitude, axial, tension–tension cyclic fatigue of continuous fiber-reinforced advanced ceramics at ambient temperatures

Table 2
Task groups within ASTM C28.07

Task group	Activity
C28.07.01	Tension
C28.07.02	Compression
C28.07.03	Creep
C28.07.04	Flexure
C28.07.05	Shear
C28.07.06	Cyclic fatigue
C28.07.07	Fibers
C28.07.08	Interfacial
C28.07.09	Thermal
C28.07.10	Environmental
C28.07.11	Thermomechanical fatigue
C28.07.12	Structural/components

Table 3
Industry-accepted, non-consensus standards developed by NASA's Enabling Propulsion Materials Program

HSR/EPM-TSS-001-93	Measurement of test system alignment under tensile loading
HSR/EPM-D-001-93	Consensus standard, monotonic tensile testing of ceramic matrix, intermetallic matrix and metal matrix composites
HSR/EPM-D-002-93	Consensus standard, tension–tension load controlled fatigue testing of ceramic matrix, intermetallic matrix and metal matrix composites
HSR/EPM-D-003-93	Consensus standard, four-point flexure testing of ceramic matrix, intermetallic matrix and metal matrix composites
HSR/EPM-D-004-93	Consensus standard, creep rupture and stepped-creep rupture of ceramic matrix, intermetallic matrix and metal matrix composites
HSR/EPM-NDE-001-93	Consensus standard, measurement of the bow and warp of continuous fiber-reinforced test specimens
Proposed	CMC pre-cracking standard

two-dimensionally reinforced (2-D) CFCCs it recommends the use of specimens with contoured gauge sections. This document also addresses the need to test specimens having dimensions (e.g., volume) that are consistent with the ultimate use of the tensile data. Fig. 2 shows examples of recommended test specimen geometries in C1275.

Applied force is transferred to the specimens through gripping devices. Gripping devices can have either active (e.g., hydraulically-actuated grips) or passive (e.g., edge- or pin-loaded arrangements) interfaces, and the availability of a given type of testing arrangement will dictate the type of specimen geometry and vice versa.

Gripping devices are typically attached to the test system through couplers, which can be classified either as fixed or non-fixed. However, regardless of the type of coupler used, C1275 mandates verification of the alignment of the test system either prior to each test, or before and after a series of tests. Analytical and empirical studies have concluded that, for negligible effects on the estimates of the strength distribution parameters for monolithic ceramics, allowable percentage bending as defined in ASTM Practice E1012 should not exceed 5% [5,6]. Although recent studies have revealed that the ultimate tensile strength of some CFCCs is relatively insensitive to percentage bending [5],[2] in the absence of more complete studies for CFCCs C1275 adopted the same percentage bending requirements as for tensile testing of advanced monolithic ceramics.

In contrast to the apparent insensitivity of the ultimate tensile strength of CFCCs to percentage bending, the so-called proportional stress limit, which is associated with the onset of matrix cracking,[3] is very sensitive to percentage bending [7]. This is important because, in the absence of environmentally stable fibers and fiber coatings, it appears that the proportional stress limit will be considered an upper limit for design stresses for many CFCCs. Therefore, to accurately determine the proportional stress limit of CFCCs, it is essential to meet the allowable bending strain requirements prescribed in C1275.

Test standard C1275 also addresses different techniques for strain measurements, such as optical methods using lasers and flags, or contact methods such as adhesively bonded strain gauges and extensometers. However, regardless of the type of extensometer used, this shall satisfy Class B-1 requirements as outlined in Practice E83 [8]. Additional requirements for contact-type extensometers are that the extensometer should not damage the specimen, and to be externally supported so that its weight does not introduce bending strains greater than those allowed in C1275.

[2] This apparently results from loading large aspect ratio fibers when these bridge matrices crack in the composite.

[3] Actually, matrix microcracking occurs at stresses lower than the proportional stress limit, but environmental sensitivity, which results from ingress of the environment into the composite, is mostly associated with this macroscopic stress.

As indicated, test standard C1275 became the template for all other ASTM test standards for CFCCs involving tensile testing. These standards, particularly those addressing tests at elevated temperatures, have special provisions in addition to the requirements outlined in C1275. These include, for example, issues such as heating methods and temperature measurements, and address the importance of mechanical testing rates, since many CFCCs may exhibit time-dependent deformation at elevated temperatures.

All of the existing tensile-based ASTM standard test methods for CFCCs address conducting tests in ambient air, but given the importance of environmental effects on the mechanical behavior of CFCCs, ongoing work has been focused on the drafting of test methods to conduct tests in controlled simulated environments.

2.2. Shear testing

Except for the torsion testing of thin-walled tubes, there is no single ‘good’ test to measure the shear strength of CFCCs [9]. Because the preparation and testing of tubular specimens to measure the shear strength of CFCCs would be prohibitively expensive, standard test method C1292 for Shear Strength of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperatures addresses two popular, but less than perfect, test methods for determining the shear strengths of uni-directionally and two-directionally reinforced CFCCs: the compression of a double-notched specimen to determine interlaminar shear strength, and the Iosipescu test to determine both in-plane and interlaminar shear strength. Schematics of the specimen geometries and test configurations for these two tests are shown in Fig. 3. Since both of these tests have been widely used for the evaluation of polymer matrix composites, much of the groundwork for the drafting of C1292 had already been laid down.

Although both the compression of double-notched specimens and the Iosipescu test have the advantage of requiring relatively small specimens and being simple to conduct, their main disadvantage is that both rely on the stress concentration at the root of notches to initiate shear failure. As a consequence, in the case of the compression of double-notched specimens, for example, the shear stress in the gauge section (the region between notches) is not uniform, and furthermore the apparent interlaminar shear strength depends on the distance between the notches [10]. Nevertheless, these test methods provide conservative shear strength values.

2.3. Flexure testing

One of the motivations for developing a standard test method to evaluate CFCCs in tension was attributed to the discrepancies between, and the wide range of, ‘strength’ values reported when CFCCs were evaluated in flexure. The problem is that, when testing CFCCs in flexure, it is difficult to relate the forces and displacements measured

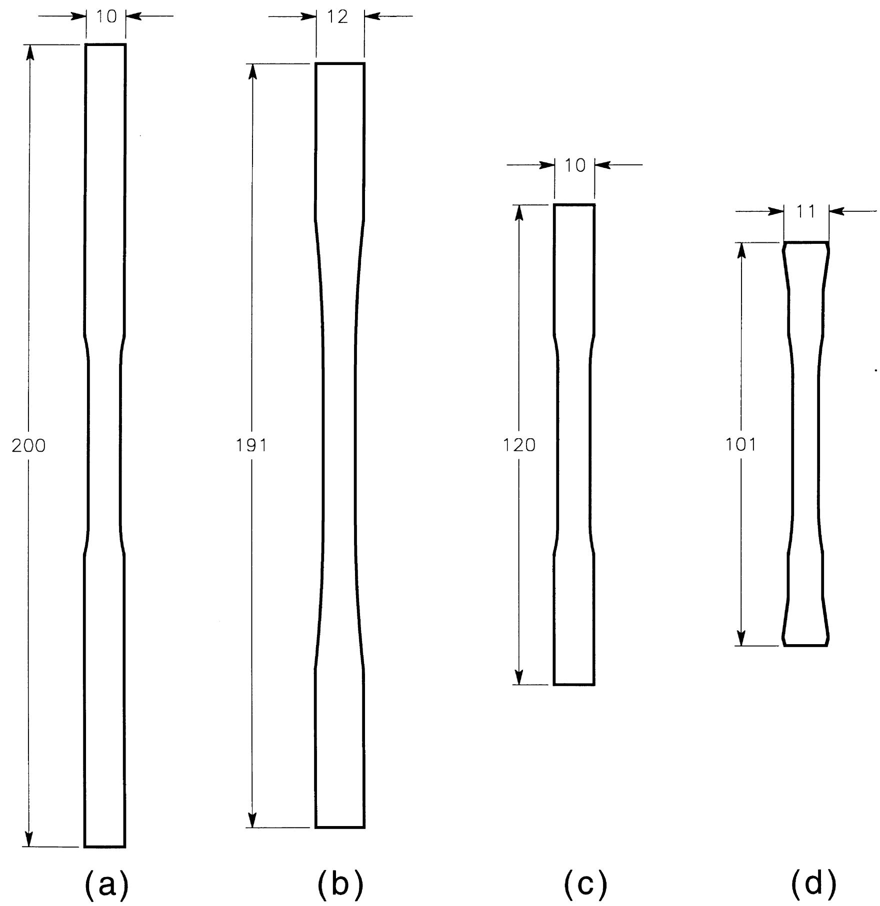

Fig. 2. Examples of tensile specimen geometries recommended in ASTM C1275: (a–c) face-loaded specimens; (d) shoulder-loaded specimen. Typical dimensions in mm.

during the test to key material properties (e.g., tensile strength, work of fracture) through a simple analysis. As a result, equations that were derived for linear elastic, isotropic, homogeneous materials that exhibit symmetric behavior in tension and compression, and that have been customarily used to calculate the so-called 'flexural strength' of CFCCs, are not applicable for these materials. In spite of this, flexural testing has been and remains a popular test method in industrial laboratories because of its simplicity and because it requires relatively small samples. As a result of the interest expressed by industry to continue using this method, standard test method C1341 (Standard Test Method for Flexural Properties of Continuous Fiber-Reinforced Advanced Ceramics) was developed and approved in 1996. The scope of C1341 is limited to the use of 'flexural data' for quality control and material development and, in contrast to flexural data for monolithic ceramics, the use of CFCC flexure data for design purposes is discouraged.

2.4. Other

Recently, the jurisdiction of D3379 (Test Method for Tensile Strength and Young's Modulus for High-Modulus Single-Filament Materials) was transferred from ASTM committee D30 to subcommittee C28.07. This coincided with ongoing efforts of a task group in C28.07 to develop a standard test method to determine the tensile properties of ceramic fibers both at room and elevated temperatures. Although there are still issues pending resolution (e.g., fiber diameter measurements), it is expected that a revision of D3379 will include much of the work developed by task group C28.07.07.

Other documents dealing with the thermomechanical behavior of CFCCs, the strength of CFCC–CFCC joints, the tensile transverse strength of CFCCs, the determination of CFCC fiber–matrix interfacial properties and the hoop strength of CFCC tubular components are currently in draft form or undergoing the ASTM balloting process, and it is expected that these will become ASTM standards in the near future.

2.5. Round robin

The ASTM requires that standard test methods include precision and bias statements. Because of the lack of

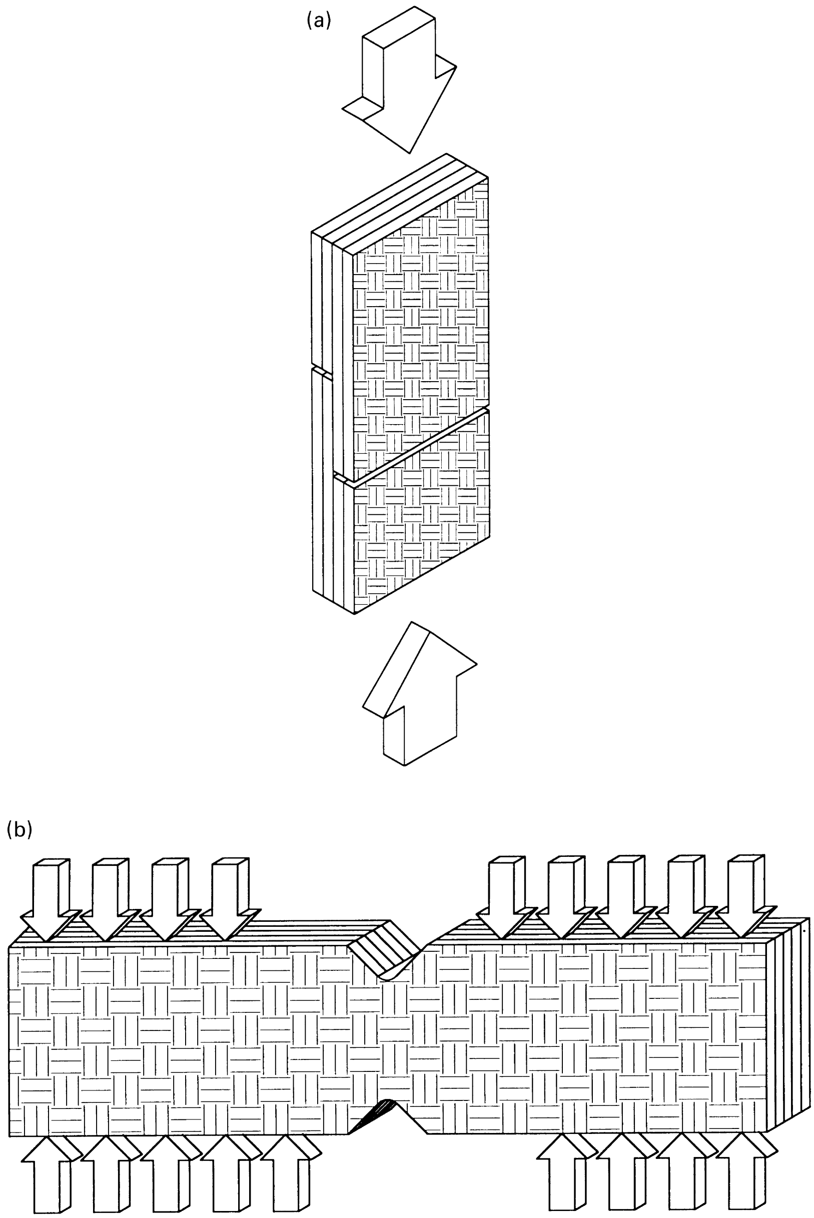

Fig. 3. (a) Schematic of compression of double-notched specimen for the determination of interlaminar shear strength of CFCCs. (b) Schematic of Iosipescu specimen for the determination of in-plane shear properties of CFCCs.

extensive databases with mechanical properties of CFCCs, existing ASTM standards for CFCCs lack these statements, but must include them during their next revision. The US Department of Energy sponsored program on Continuous Fiber-Reinforced Ceramic Composites recently sponsored a round robin testing program to determine the precision and bias statements for standard test methods C1275, C1292 and C1341. In addition to fulfilling this goal, the round robin testing will produce an expanded database for the material that will be used in the study, along with statistical distributions of properties and performance for both design and production purposes. More than 10 laboratories, including national laboratories, material manufacturers, universities and independent research institutes, will participate in this effort, which is scheduled to be completed by the Spring of 1998.

2.6. International harmonization

The US is represented at the technical committees (TCs) of the International Organization for Standardization (ISO) through technical advisory groups (TAGs) of the American National Standards Institute (ANSI). In 1994, ANSI named the ASTM as administrator of TAG-206 to represent the US at ISO's TC206 on Fine (Advanced, Technical) Ceramics. In February 1997, a working group (WG9) on tensile behavior of CFCCs was officially established as part of TC206, with the US serving as the convenor. At the first official meeting of WG9 in July 1997, work focused on refining the working group's draft for elevation to, first, committee draft (CD), and then to draft an international standard (DIS) in preparation for its establishment as the first ISO standard for CFCCs. The significance of ISO standards is tremendous when one considers that disputes between countries that are members of the General Agreement for Tariffs and Trade (GATT) will be resolved using ISO standards.

ASTM subcommittee C28.07 has established, and maintains, strong liaisons with other international standardization organizations, such as the Comité Européen de Normalisation (CEN) Technical Committee 184. As part of the agreement of understanding between ASTM C28.07 and CEN TC184 subcommittee SC1 on Ceramic Matrix Composites, officially initiated in September 1996, mechanisms have been established for the exchange of documents, for the organization of joint round robin efforts and technical meetings, and for the synchronization of efforts towards the development of ISO standards.[4]

3. Design codes

Design codes not only qualify measurements of material properties for inclusion in databases or handbooks (often requiring the use of existing standards), but also provide design methodologies for using these unique materials. Thus, the current efforts in the development of design codes in the US may represent the culmination of the standardization process for CFCCs.

In 1995, an effort was initiated to draft a code for the use of CFCCs and ceramics in pressure equipment. The relevance of this activity stems from the fact that certification of pressure equipment is a legal requirement in 48 states of the US.

The objective of this effort was to introduce impervious graphite and ceramic composites as acceptable performance-based materials in the ASME Boiler and Pressure Vessel Code. Therefore, a subtask group on Ceramic Pressure Equipment was organized as part of the task group on Graphite and Ceramic Pressure Equipment of the ASME Boiler and Pressure Vessel Code. The proposed sections of the code for CFCCs include: general, material, design, fabrication, qualification, pressure relief, rules governing testing, inspection, and marking and stamping.

At the same time, there is an ongoing effort, initiated in 1996, to draft a set of volumes on ceramic matrix composites for Military Handbook 17. This is a set of volumes with the primary purpose of standardizing methodologies for generation of engineering design data related to testing, data reduction and data reporting for current and emerging composite materials. The organization of the work towards the drafting of a CFCC section for Military Handbook 17 is schematically represented in Fig. 4. The Military Handbook

[4] The European Community has no representation at the ISO.

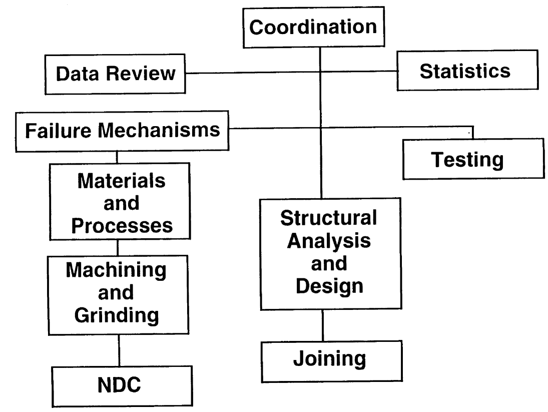

Fig. 4. Organization of Military Handbook 17 activities for ceramic matrix composites.

17 effort was initiated in 1959 for polymer matrix composites. Later, an effort for metal matrix composites was initiated in 1993.

4. Summary

Since its establishment in 1991, ASTM subcommittee C28.07 on ceramic matrix composites has actively promoted the development of standardized test methods for CFCCs and has been responsible for formalizing seven full consensus standard test methods for CFCCs and for drafting several other documents that are currently going through the internal ASTM balloting process. Current efforts in the ASME and Military Handbook 17 toward the development of design codes in the US may not only represent the culmination of the standardization process for CFCCs, but also certainly mark the first steps in the development of reliable databases and guidelines for designing components using CFCCs.

Acknowledgements

This work was sponsored by the US Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Industrial Energy Efficiency Division, Continuous Fiber-Reinforced Ceramic Composites Program under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corporation. The authors are grateful to their colleagues E.Y. Sun, K.C. Liu and Arvid Pasto of ORNL, and M. Taya of the University of Washington, for reviewing the manuscript.

References

[1] Karnitz MA, Craig DF, Richlen SL. Continuous fiber ceramic composite program. Ceramic Bulletin 1991;70(2):430–435.

[2] Quinn GD. ASTM C-28: the first decade. Ceramic Bulletin 1996;75(12):45–46.
[3] Cranmer C. Workshop on assessment of testing methodology for glass, glass-ceramic, and ceramic matrix composites. Journal of Research of the National Institute of Standards and Technology 1991;96(4):493–501.
[4] Jenkins MG. CFCC Program Task 2: Supporting Technologies Progress Report for July 1992. Oak Ridge National Laboratory, 1992:22–27.
[5] Jenkins MG, Ferber MK, Martin RL, Jenkins VT, Tennery VJ. Study and analysis of stress state in a ceramic button-head tensile specimen. Oak Ridge National Laboratory Technical Memorandum, ORNL/TM 11767, September 1991.
[6] ASTM Practice E1012. Practice for Verification of Specimen Alignment under Tensile Loading, Annual Book of ASTM Standards, Vol. 03.01.
[7] Jenkins MG, Piccola JP, Lara-Curzio E. Influence of bending, test mode, test rate, specimen geometry and grip system on the tensile mechanical behavior of CFCCs. In: Jenkins MG, Gonczy ST, Lara-Curzio E, Ashbaugh NE, Zawada LP, editors. Thermal and mechanical test methods and behavior of continuous fiber ceramic composites (ASTM STP 1309). W. Conshohocken, PA: American Society for Testing and Materials, 1997.
[8] ASTM Practice E83. Practice for verification and classification of extensometers. Annual Book of ASTM Standards, Vol. 03.01.
[9] Lara-Curzio E, Ferber MK, Jenkins MG. Methodologies for the thermomechanical characterization of continuous fiber-reinforced ceramic matrix composites: a review of test methods. In: Proceedings of the 39th International SAMPE Symposium, Anaheim, CA, 11–14 April 1994:1780–1789.
[10] Lara-Curzio E, Ferber MK. Shear strength of continuous fiber reinforced ceramic composites. In: Jenkins MG, Gonczy ST, Lara-Curzio E, Ashbaugh NE, Zawada LP, editors. Thermal and mechanical test methods and behavior of continuous fiber ceramic composites, ASTM STP 1309. W. Conshohocken, PA: American Society for Testing and Materials, 1997.