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Standards and codes for ceramic matrix composites

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Standards and codes for ceramic matrix composites

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Abstract—Ceramic matrix composites (CMCs) and, in particular, continuous fibre ceramic composites (CFCCs) are targeted for industrial, aerospace and other high-technology applications that require the high-temperature properties and the wear/corrosion resistance of advanced ceramics while providing inherent damage tolerance (i.e. increased 'toughness') without the volume/surface area-dependent strengths of monolithic ceramics. To utilize CFCCs designers need reliable and comprehensive data bases (and the design codes that contain them). Generating reproducible information for these data bases requires standards. Presently, there are relatively few (compared to metals) national (e.g. ASTM, CEN, JIS, etc.) or international standards (e.g. ISO) for testing CFCCs. In this paper, the various standards for CFCCs are reviewed and additional areas requiring normalization are discussed (e.g. mechanical, thermal, electrical, electro-magnetic, optical, and biological testing). 'Design codes' such as the ASME Boiler and Pressure Vessel Code discussed here, are widely accepted, general rules for the construction of components or systems (for performance, efficiency, usability, or manufacturabilty) with emphasis on safety. Wide-ranging codes incorporate figurative links between materials, general design, fabrication techniques, inspection, testing, certification, and finally quality control to insure that the code has been followed. Implicit in design codes are many of the standards for materials testing, characterization, and quality control. Logical outcomes of design codes are data bases of material properties and performance 'qualified' for inclusion in the code. As discussed in this paper, data bases (such as those contained in the Mil-Hdbk-17 CMC effort) may be in print, electronic or world-wide web-based formats and may include primary summary data (e.g. mean, standard deviation, and number of tests) along with secondary data (e.g. graphical information such as stress–strain curves).

Keywords: Ceramic matrix composites; standards; design codes; data bases.

1. INTRODUCTION

Thermo-mechanical behaviour (and its subsequent characterization) of ceramic matrix composites (CMCs) along with the CMC subset, continuous fiber-reinforced ceramic–matrix composites (CFCCs) is currently the subject of extensive investigation worldwide. In particular, determination of the properties and performance (mechanical, thermal, thermo-mechanical, physical, environmental, etc.) of CMCs and CFCCs is required for a number of reasons: (1) to provide basic characterization for purposes of materials development, quality control and comparative studies; (2) to provide a research tool for revealing the underlying mechanisms of thermal and mechanical performance; and (3) to provide engineering performance-prediction data for engineering applications and components design [1]. As CFCC prototype and trial products begin to reach the marketplace, the paucity of standards (i.e. test methods, classification systems, unified terminology, and reference materials) for these materials and the lack of CFCC design codes and their related data bases are limiting factors in commercial diffusion and industrial acceptance [2] of these advanced materials.

Standards. The term 'standards' has many implications. To the researcher and the technical community it may be fundamental test methodologies and units of measure. To the manufacturer or end-product user it may be materials specifications and tests to meet requirements. Commercial standards equate to the rules and terms of information transfer among designers, manufacturers and product users [2]. There are even fundamental differences between levels of standards: company (internal use with only internal consensus); industry (trade/project use with limited organizational consensus); government (wide usage and varying levels of consensus); full-consensus (broadest usage and greatest consensus).

At present, there are few — nationally or internationally — full-consensus standards [3–5] for testing not only advanced ceramics but especially CFCCs. This limited ability to test on a common-denominator basis hampers further material development [2]. Specific areas where standardization (or consensus) are required include: test fixtures, test specimen geometries, specimen preparation, machining procedures and allowable tolerances, test specimen alignment, optimal straining/stressing rates, metrology (temperature and strain), testing environment and identification of fracture and failure modes. These needs are particularly acute at elevated temperatures or in aggressive environments where test equipment and measurement techniques are often being developed simultaneously with the test material. Although considerable development may be required for standards for CMCs and CFCCs, rather than adopting entirely new or unconventional methods and techniques, test methods developed originally for the room temperature characterization of polymer matrix composites (PMCs) are a good starting point to develop test methodologies for CMCs and CFCCs in particular [6].

Design codes and data bases. The meaning of the term 'design code' is not generally well understood. As used in the following discussion 'design code' is not a design manual (i.e. a 'cookbook' design procedure resulting in a desired component or system). Instead, 'design codes' are widely-accepted but general rules for the construction of components or systems with emphasis on safety. A primary objective is the reasonably certain protection of life and property for a reasonably long safe-life of the design. Although needs of the users, manufacturers and inspectors are recognized, the safety of the design can never be compromised.

By not imposing specific rules for design, codes allow flexibility for introducing new designs as required for performance, efficiency, usability, or manufacturability while still providing constraints for safety. Codes must be wide ranging, incorporating figurative links between materials, general design (formulas, loads, allowable stress, permitted details), fabrication techniques, inspection, testing, certification by stamping and data reports, and finally, quality control to insure that the code has been followed. Thus, implicit in design codes may be many of the standards previously discussed for materials testing, characterization, and quality control. In addition, unlike standards which provide no rules for compliance or accountability, codes require compliance through documentation, and certification accountability through inspection and quality control.

A logical outcome of design codes is the incorporation of data bases of material properties and performance 'qualified' for inclusion in the codes. These data are 'qualified' because they have been attained through testing per the statistical requirements of the codes as well as per the standards indicated in the codes. 'Qualified' data bases often require a minimum numbers of tests for (1) a particular batch of material and (2) multiple batches of material. In addition, data bases may include primary summary data (e.g. mean, standard deviation, and numbers of tests) along with secondary data from the individual tests. Some data bases may contain only numerical information while others may include graphical information (e.g. stress–strain curves, temperature profiles, or test specimen geometries). Data bases are increasingly in electronic form to speed data retrieval and many are even world-wide web-based to provide instant access and frequent updatability.

Design codes and their data bases may be backed as legal requirements for implementing an engineering design (e.g. certification and compliance with the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code is a legal requirement in 48 of the 50 United States). At present there are no national or international design codes allowing CFCCs in any type of design. This situation may be hampering material utilization since designers cannot use a material directly in new designs, but instead must (1) show evidence that the material meets the requirements of the code and (2) obtain special permission to use the material in the code design. In addition, material development is impaired since without a demand for a new material, there is no incentive for further refinement.

This paper concentrates on standards and codes for CFCCs. First, current standards for CFCCs are briefly reviewed keying on important mechanical, thermal, and physical aspects of testing. Next, a similar brief review of current design codes and evolving data bases for CFCCs is presented. Finally, the summary and conclusion section recaps successes and lessons and indicates future directions for standards and codes for CFCCs.

2. STANDARDS

Although the number of standards (compared to metals and metal alloys) for CFCCs is limited, this number increases with each year as this relatively new material system matures (serious concerted research dates to the mid-1970s). Table 1 shows

Table 1.
Current full-consensus standard test methods for CFCCs

Organization	Title	Completed
ASTM*	Committee C28 on 'Advanced Ceramics'	Approved
C1275-95	Standard Test Method for Monotonic Tensile Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross Sections at Ambient Temperatures	1995
C1292-95	Standard Test Method for Shear Strength of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperatures	1995
C1337-96	Standard Test Method for Creep and Creep Rupture of Continuous Fiber-Reinforced Advanced Ceramics under Tensile Loading	1996
C1341-96	Standard Test Method for Flexural Properties of Continuous Fiber-Reinforced Advanced Ceramics	1996
C1358-97	Standard Test Method for Monotonic Compressive Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross Sections at Ambient Temperatures	1997
C1359-97	Standard Test Method for Monotonic Tensile Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross Sections at Elevated Temperatures	1997
C1360-97	Standard Practice for Constant-Amplitude, Axial, Tension-Tension Cyclic Fatigue of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperatures	1997
D3379-75	Standard Test Method For Tensile Strength And Young's Modulus For High-Modulus Single-Filament Materials	1997 (C28 jurisdiction)
CXXXX**	Standard Test Method for Tensile Hoop Strength of Continuous Fiber-Reinforced Advanced Ceramic Tubular Specimens at Ambient Temperature	1998 (expected)
CXXXX**	Standard Test Method for Interlaminar Shear Strength of Continuous Fiber-Reinforced Advanced Ceramics at Elevated Temperatures	1998 (expected)
CXXXX**	Standard Test Method for Transthickness Tensile Strength of Continuous Fiber-Reinforced Advanced Ceramics Ambient Temperature	1999 (expected)
CXXXX**	Standard Test Method for Tensile Strength and Young's Modulus for High-Modulus Single Filament Advanced Ceramics	1999 (expected)
CXXXX**	Standard Test Method for Asymmetric Four-Point Shear Strength of Ceramic Joints in Continuous Fibre-Reinforced Advanced Ceramics at Ambient Temperature	1999 (expected)
CEN*	Committee TC184 'Advanced Technical Ceramics'	Approved
ENV 658-1	Tensile Strength of Continuous Fibre Reinforced Ceramic Composites	
ENV 658-2	Compressive Strength of Continuous Fibre Reinforced Ceramic Composites	
ENV 658-3	Flexural Strength of Continuous Fibre Reinforced Ceramic Composites	
ENV 658-4	Shear Strength (compression) of Continuous Fibre Reinforced Ceramic Composites	

Table 1.
(Continued)

ENV 658-5	Shear Strength (3-point) of Continuous Fibre Reinforced Ceramic Composites	
ENV 658-6	Shear Strength (double punch) of Continuous Fibre Reinforced Ceramic Composites	
ENV 1159-1	Thermal Expansion of Continuous Fibre Reinforced Ceramic Composites	
ENV 1159-2	Thermal Diffusivity of Continuous Fibre Reinforced Ceramic Composites	
ENV 1159-3	Specific Heat of Continuous Fibre Reinforced Ceramic Composites	
ENV 1389	Density of Continuous Fibre Reinforced Ceramic Composites	
ENV 1007-1	Size Level of Fibres for Continuous Fibre Reinforced Ceramic Composites	
ENV 1007-2	Linear Mass of Fibres for Continuous Fibre Reinforced Ceramic Composites	
ENV 1007-3	Filament Diameter of Fibres for Continuous Fibre Reinforced Ceramic Composites	
ENV 1007-4	Filament Strength of Fibres for Continuous Fibre Reinforced Ceramic Composites	
ISO*	Committee TC206 'Fine (Advanced Technical) Ceramics'	
CD 15733	Test Method for Tensile Stress-strain Behaviour of Continuous Fibre-reinforced Composites at Room Temperature	1998

* ASTM: American Society for Testing and Materials; CEN: Committee for European Normalization; ISO: International Organization for Standardization.

** Draft standard in the ballot process.

current full-consensus standards for CFCCs (American Society for Testing and Materials (ASTM), Committee for European Normalization (CEN), International Organization for Standardization (ISO)). Table 2 lists industry/government standards for CFCCs (Enabling Propulsion Materials (EPM), Petroleum Energy Center (PEC)). The following subsections provide brief overviews of some of the key issues in standards for tension, compression, shear, flexure, constituents, physical, and other areas. It is important to realize that although no component fabricated from CFCC will always be subjected to uniaxial, uniform stress states, unambiguous interpretation of test results for characterization of the mechanical response of CFCCs requires well-developed and understood stress fields such as uniaxial tension or compression. In particular, CFCCs often show different responses in tension and compression (see Fig. 1) which complicate analysis of results from the tests with non-uniform stresses such as flexure [7, 8]. In addition, although many of the standards developed to date have been for ambient conditions, CFCCs are targeted for elevated temperatures in aggressive environments which compound already stringent testing demands.

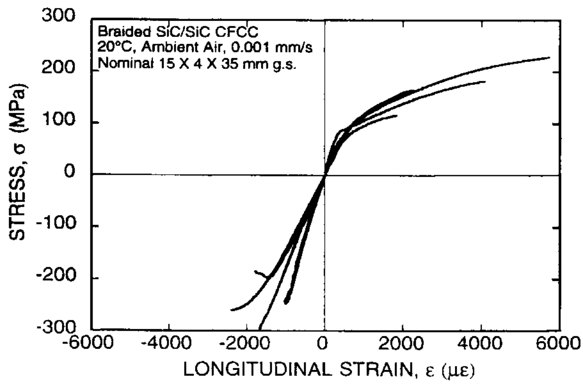


Figure 1. Differences in response of a CFCC in tension and compression [8].

2.1. Tension

Uniaxial tensile testing is the most fundamental means of measuring mechanical response of most engineering materials and interest in it for CFCCs is driven primarily by limitations of the flexure tests widely used to characterize monolithic ceramics. However, because of the difficulty of deconvoluting tensile response from the flexure results, a uniaxial tensile test is the preferred tensile test for the response of CFCCs in the plane of the reinforcement. This preference has led to a wide range of test specimen geometries (straight-sided to contoured) and gripping systems (face, pin or edge 'loaded') as illustrated in Fig. 2. Another issue which must be addressed for CFCCs is the material's sensitivity to non-uniform stress (i.e. bending during the tensile test) (see Fig. 3) [9]. The mode (force, displacement, or strain control) and rate (rapid or slow) of testing as well as means of strain measurement (optical, contacting, etc.) are important concerns as well, especially at elevated temperature at which creep or other time-dependent mechanisms may be operative.

Because the uniaxial tension test is the most fundamental test for mechanical characterization, the greatest number of in-plane monotonic tensile test standards (see Tables 1 and 2) exist for it (two ASTM, three CEN (standards or advanced drafts), one ISO, one EPM, one PEC). Once developed, the monotonic tensile test also becomes the basis for other types of tests, such as cyclic fatigue or creep/creep rupture. Recent efforts in both ASTM and CEN are moving toward developing in-plane, elevated-temperature tensile tests for various environments other than ambient air. Concerns here are the type of grip (hot, warm, or cold) and the design of the test specimen so as to minimize the imposition of undesirable thermal and imposed stresses (see Fig. 4) [8].

More recently, demand has increased to determine the tensile response perpendicular to the in-plane reinforcements (see Fig. 5). In two-directionally reinforced CFCCs, this direction can be the weakest since only matrix material with some interphase and no reinforcing fibres are present. An ASTM document on 'trans-

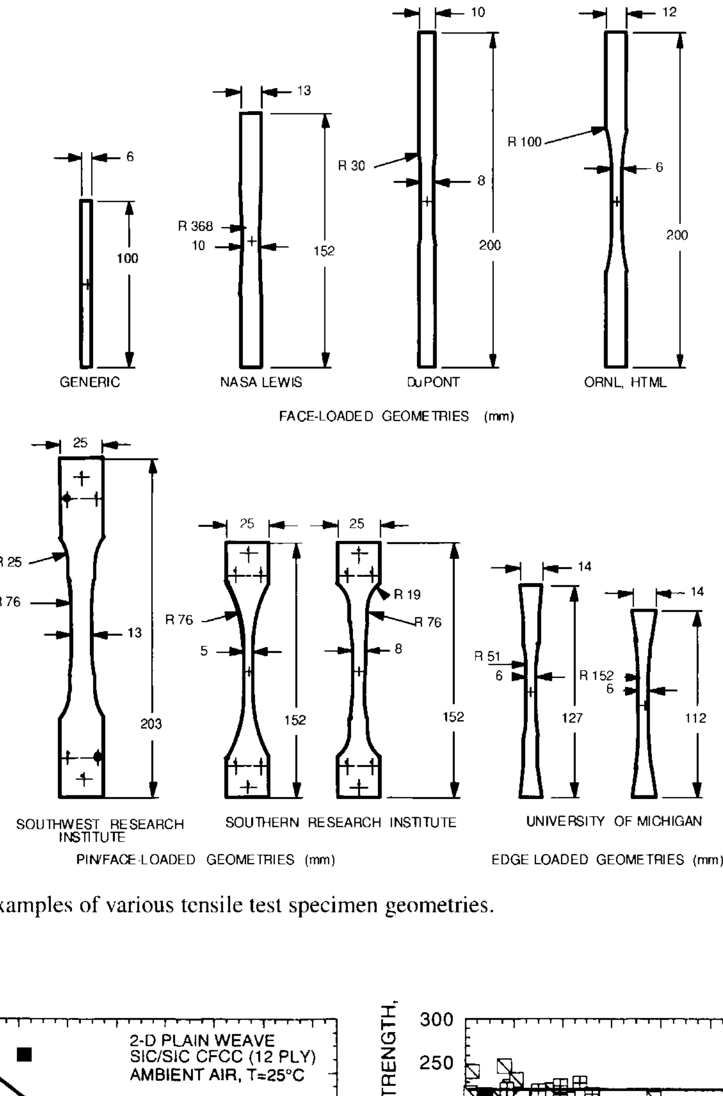
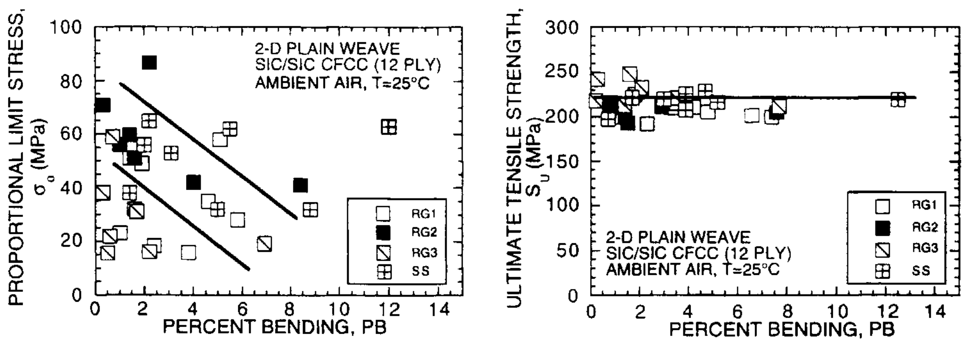


Figure 2. Examples of various tensile test specimen geometries.



a) Proportional limit stress

b) Ultimate tensile strength

Figure 3. Strength as a function of percent bending in room temperature monotonic tensile tests (a) Proportional limit stress and (b) Ultimate tensile strength [8].

Table 2.
Current industry/government standard test methods for CFCCs

Organization	Title	Completed
EPM* (USA)		Approved
HSR/EPM-D-001-93	Monotonic Tensile Testing of Ceramic Matrix, Intermetallic Matrix and Metal Matrix Composite Materials	1993
HSR/EPM-D-002-93	Tension-tension Load Controlled Fatigue Testing of Ceramic Matrix, Intermetallic Matrix and Metal Matrix Composite Materials	1993
HSR/EPM-D-003-93	Four Point Flexure Testing of Ceramic Matrix, Intermetallic Matrix and Metal Matrix Composite Materials	1993
HSR/EPM-D-004-93	Creep-Rupture and Stepped Creep Rupture of Ceramic Matrix, Intermetallic Matrix and Metal Matrix Composite Materials	1993
HSR/EPM-TSS-001-93	Measurement of Test System Alignment Under Tensile Loading	1993
HSR/EPM-NDE-001-93	Measurement of the Bow and Warp of Continuous Fiber Reinforced Test Specimens	1993
Task A.5.4/A.5.5	CMC Pre-Cracking Standard	—
PEC* (Japan)		Approved
PEC-TS CMC01	Test Method for Tensile Stress-Strain Behaviour of Continuous Fibre Reinforced Ceramic Matrix Composites at Room and Elevated Temperatures	1997
PEC-TS CMC04	Test Method for Flexural Strength of Continuous Fibre Reinforced Ceramic Matrix Composites at Room and Elevated Temperatures	1997
PEC-TS CMC06	Test Method for Shear Strength of Continuous Fibre Reinforced Ceramic Matrix Composites at Room and Elevated Temperatures	1997
PEC-TS CMC08	Test Method for Fracture Toughness of Continuous Fibre Reinforced Ceramic Matrix Composites	1997
PEC-TS CMC09	Test Method for Fracture Energy of Continuous Fibre Reinforced Ceramic Matrix Composites	1997
PEC-TS CMC010	Test Method for Tensile-Tensile Cyclic Fatigue of Continuous Fibre Reinforced Ceramic Matrix Composites at Room and Elevated Temperatures	1997
PEC-TS CMC011	Test Method for Tensile Creep of Continuous Fibre Reinforced Ceramic Matrix Composites at Elevated Temperatures	1997
PEC-TS CMC013	Test Method for Elastic Modulus of Ceramic Matrix Composites at Room and Elevated Temperatures	1997
PEC-TS CMC014	Test Method for Oxidation Resistance of Non-Oxide Ceramic Matrix Composites at Elevated Temperatures	1997

*EPM = Enabling Propolusion Materials program (NASA, GE, Pratt and Whitney consortium) (USA). PEC = Petroleum Energy Center (Japan).

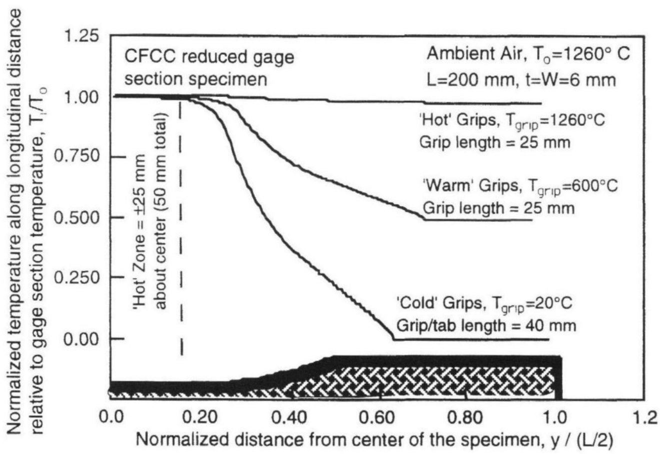


Figure 4. Temperature distributions in a contoured tensile test specimen for various types of grip cooling arrangements [7].

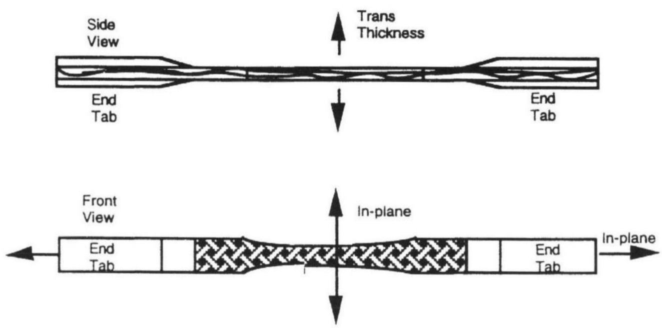


Figure 5. In-plane and trans thickness directions for a 2D CFCC.

thickness' tensile strength is currently undergoing the balloting process to address this concern.

2.2. *Compression*

While it is accepted that elastic moduli for CFCCs measured from compressive tests are independent of the test method, compressive strength results depend on the mode of failure and hence, the compression test method employed. Regardless of the method used, compressive performance is the most difficult to evaluate because of test scatter greater than that of any other test. The major source of scatter is differences in failure modes [10], while the major experimental difficulties for compression tests are buckling instabilities and achieving good alignment to avoid load eccentricities that induce bending.

To avoid brooming and bending, robust tabs are usually bonded to the uniaxially loaded specimens. Buckling resistance can be increased if clamped end conditions are achieved and the bending stiffness near the ends is increased [10]. Finite element analyses (FEA) have been employed to optimize the loading configuration of compression test specimens which are based upon: preventing overall buckling, preventing debonding of end tabs and preventing failure at the gage region-tab juncture while insuring stress uniformity in the central region [10]. Standardized compression tests for CFCCs have tended toward uniaxially-loaded, unsupported test specimens (i.e. no antibuckling guides) in anticipation of elevated temperature tests. Results (see Fig. 6) from these compression tests have been compared to tension and flexure test results to illustrate similarities (elastic modulus) and differences (strengths) between the different types of tests.

Another test available to determine the compressive performance of composites is the bending of sandwich beams. If the face in tension is deliberately made much stronger than the face in the compression side, a compressive failure will be induced. This test provides effective load transfer, ideal compressive strengths and data comparable to uniaxially loaded compression tests making the bending of composite sandwich beams highly recommended for design allowables. The major disadvantages for this test are that the test specimen is quite large and that the bonded core may artificially enhance the performance of the face in compression, although round-robin tests with graphite/epoxy composites have alleviated the latter concern [11]. Limited data are available in the open literature [8, 12] on compressive strength of CFCCs, although the standards currently in place (see Tables 1 and 2) should provide reliable and reproducible test results.

2.3. *Shear*

The three primary testing modes are tension, compression and shear. There are three components of tensile and compressive stresses: axial, in-plane transverse and through the thickness transverse. Correspondingly, there are also three components of shear stresses: in-plane and two through-thickness components. Most current shear tests methods measure in-plane properties and only a few measure the interlaminar or through-thickness shear. Correspondingly, there are tests that only provide data on strength, or stiffness or both [11].

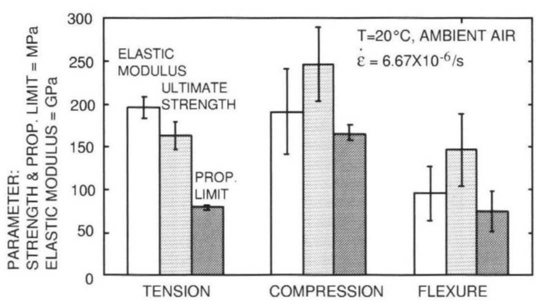


Figure 6. Bar chart comparisons of elastic modulus, ultimate strength, and proportional limit at 20°C for tension, compression and flexure for a 3D braided CFCC [7].

The ideal quantitative shear test method should provide a region of pure and uniform shear stress. In addition, there should be a unique relationship between the applied load and the magnitude of the shear stress in the test section, and the test section should be one of maximum shear stress relative to all other regions of the specimen. Although it can be argued that there are no good shear tests but only varying degrees of bad ones, the torsion of a thin tube comes closest to satisfying the criteria for a good in-plane shear test.

Nonetheless, shear test methods may be divided into two groups: those that use flat specimens and those that use tubular or circular section specimens. For CFCCs, standards (see Tables 1 and 2) that use flat specimens include (see Fig. 7): the double notch compression test and the short flexure beam for interlaminar shear behaviour and the double V-notch test (also called the Iosipescu test) for in-plane shear behaviour. Currently, there are no standards for CFCCs which use torsion for shear behaviour, although examples of tests using specimens with circular cross sections include the torsion of tubes and the four-point ring-twist test.

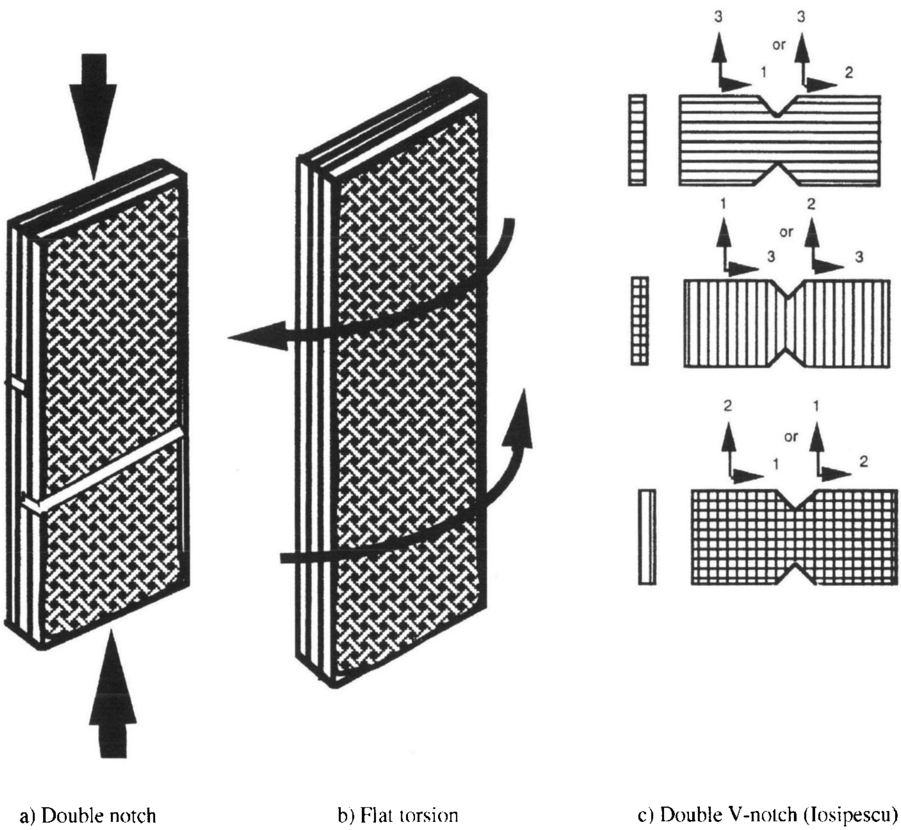


Figure 7. Shear test specimens (a) Double-notch compression, (b) Flat torsion and (c) Double V-notch (Iosipescu).

2.4. Flexure

While one could argue that no component undergoes uniform stress states such as those imposed by properly conducted tension, compression or shear tests, these tests do serve the purpose of extracting material behaviour under controlled and well-documented applied stresses. However, because of differences in tensile and compressive stress–strain responses in CFCCs (see Fig. 1), the apparent 'tensile' stress–strain response of a CFCC in flexure (i.e. inferred from linear-elastic flexure relations for homogeneous, uniform materials) can be quite different (e.g. exhibiting 'graceful' failure) compared to the tensile stress–strain response of a CFCC in tension. Thus, it is sometimes important to evaluate the flexural response of a CFCC not only for apparent 'tensile' stress–strain response in flexure but also for interlaminar shear response (for short beams). There are several standards (see Tables 1 and 2) for flexure testing CFCCs (both for flexural and shear response) although the resulting measured properties are not recommended for design purposes and thus should be used for qualitative assessment only [13].

2.5. Constituents

There are three recognized primary constituents in most CFCCs: fibre, matrix and interphase. A fourth constituent, overcoat, is recently emerging as a necessary aspect for environmental barrier coatings. Understanding the properties and performance of the constituents is useful not only for predictive modeling but also for quality control. Standards (see Tables 1 and 2) exist for evaluating fibres (often single fibres, but also tows). Standards (see Tables 1 and 2) also exist for evaluating matrices, albeit usually listed for monolithic ceramics. However, no test standards currently exist for evaluating the interphase, although efforts have been initiated in ASTM to develop a guide on how to conduct and analyze single fibre push-in/push-out tests. Another promising test method being developed is the load/unload tensile test. The size and magnitude of stress–strain hysteresis loops (see Fig. 8) can be used to extract information about residual stress state and interfacial shear strength [14]. Although this type of test has not been standardized, it does offer promise for a simple mechanical test for 'interrogating' the bulk CFCC to extract *in-situ* constituent properties and performance. No standards currently exist for evaluating coatings which pose the additional difficulty of being formed as thin layers in situ.

2.6. Physical and thermal

Physical and thermal properties and performance include density, elastic constants, thermal coefficient of expansion, thermal conductivity and other non-mechanical results. Some standards (see Tables 1 and 2) have been introduced for these properties. Often, a standard test method for a monolith is adapted to apply to CFCCs. While application of the test method to the CFCC is straight forward,

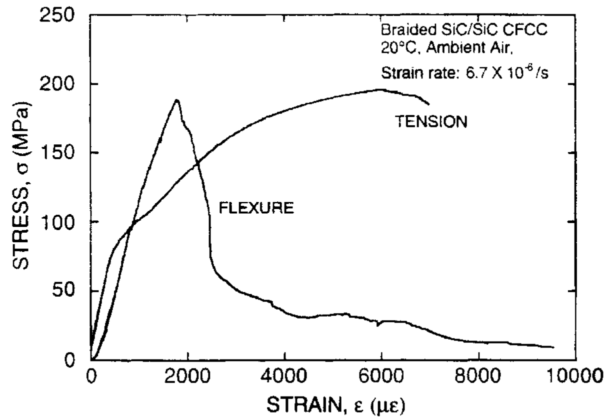


Figure 8. Comparison of engineering stress–strain curves at 20°C for uniaxial tension and apparent 'tension' in flexure [7].

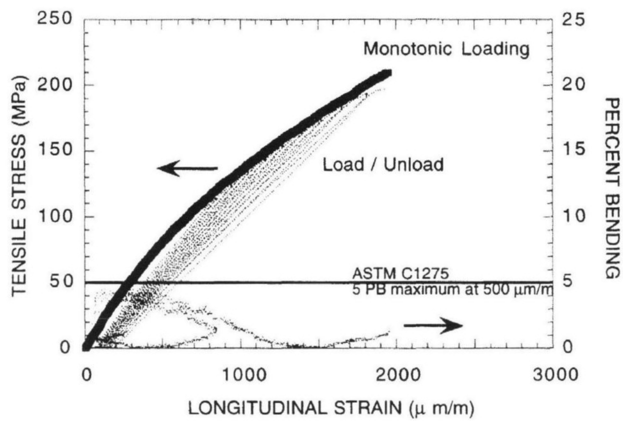


Figure 9. Comparison of stress–strain curves for monotonically-loaded and load/unload tensile tests [13].

interpretation of the resulting information is far from resolved. As attention turns from the development of standards for characterizing basic mechanical properties and performance, standards for characterizing physical and thermal aspects of CFCCs are being further developed.

2.7. *Other areas*

CFCCs are unique materials because the sum of their parts (fibre, matrix and interphase) can be greater or less than the composite itself depending on the environment or use conditions. For example, 'toughness' (i.e. area under the tensile stress–strain curve) is much greater for a CFCC than for any fibre, matrix or interphase (or their sums) because of the nonlinear energy dissipation that occurs during debonding and pullout of the fibres during the cumulative fracture process. Conversely, embrittlement and degradation of fibres is accelerated during elevated temperature exposure in air due to the presence of excess oxygen in the interphase resulting in both strength and 'toughness' of the CFCC which are less than those of any fibre, matrix or interphase (or their sums). Therefore, environmental tests (e.g. thermogravimetric analysis or differential gravimetric analysis) must be conducted under the actual use conditions. Another area needing standardization is component testing. For example, coupon test specimens cannot be extracted from CFCC tubes without disturbing the unique weaving for the fibres.

As CFCCs become accepted in the structural design community, the number and variety of applications will increase. With these applications will come the need to characterize the electrical, electro-mechanical, optical and biological aspects of CFCCs.

3. DESIGN CODES AND DATA BASES

Design codes contain in them not only the methodologies for successfully executing the steps in the design of a particular component or system, but also the information required to complete the design. This information is in the form of qualified data bases which contain material properties and performance obtained using standards referenced in the design code as well as statistical means for evaluating the 'quality' of the data, also contained in the design code. Two efforts are currently underway to establish such design codes for CFCCs: ASME Task Group on Ceramic and Graphite Pressure Equipment and Military Handbook 17 (Mil-Hdbk-17) CMC effort.

3.1. ASME Task Group on Ceramic and Graphite Pressure Equipment

The ASME Boiler and Pressure Vessel Code [15] is a good example of a design code where the material-specifics of CFCCs must be incorporated before one of the potential applications (power generation) of CFCCs can be realized. As shown in Fig. 10, the 'Code' is divided into eleven sections, the two most important for CFCCs being Section I: Power Boilers and Section II: Materials, each detailed in the following discussion.

Section I dates to the adoption of the 'Code' in 1914 and is divided into eight subgroups each dealing with specific aspects of the care, piping, design, fabrication and examination of power boilers [16]. In addition, subgroups also deal with particular types of boilers, including firetube and electric. Generally, Section I applies to boilers with $>$103 kPa pressure external to the boiler itself and $>$ 1103 kPa pressures and/or $>$ 121°C temperatures for high-temperature water boilers. The scope of jurisdiction of Section I is the boiler proper and the boiler external piping [16].

Of particular interest for CFCC producers is the subgroup on materials in Section I which limits materials to (1) those listed in Section II and (2) those listed in certain tables in Section I. In addition, certain materials are restricted in regard to usage.

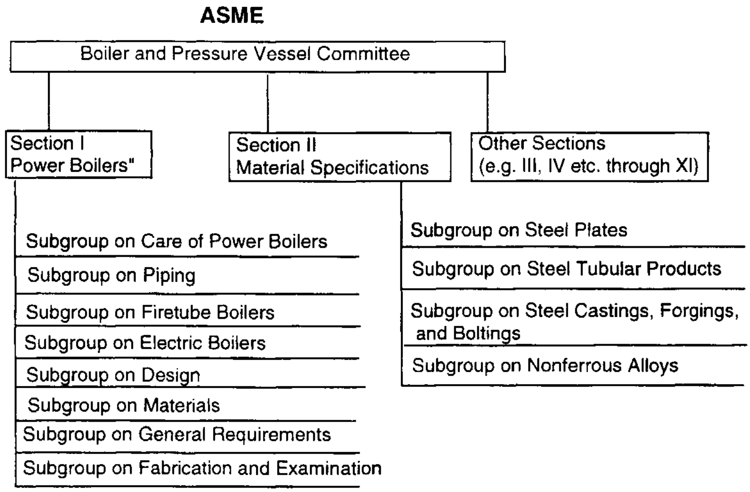


Figure 10. Partial overall view of organization of ASME Boiler and Pressure Vessel Code.

For example, an austenitic stainless steel might not be permitted for parts in water-wetted service, but may be permitted for steam-touched service. Also included in Section I, are provisions for Approval of New Materials for Code Construction [14, 15]:

(1) New materials shall have been previously adopted by ASTM (Adoption of a new materials for the ASME Code means adoption of the ASTM specification for the material).

(2) Items to be furnished for evaluation by the 'Code' committee are:

  (i) such mechanical properties as ultimate tensile strength, yield strength, creep and rupture strength, heat treatment, toughness, etc.;
  (ii) stress–strain data for vessels designed for external pressure;
  (iii) weldability, including data for establishing the requirements of Section IX: Welding;
  (iv) physical changes and resistance to effects of both elevated temperature and cryogenic temperature where applicable;
  (v) availability of the material regarding patents and licensing.

For design purposes, two approaches are prescribed in the code: design by rule and design by analysis. Section I relies primarily on design by rule which is basically an empirical approach (what has proven to work successfully in the past) setting limits on: factors of safety (typically 4–5); design pressures and temperatures, minimum thicknesses (e.g. 6.35 mm); maximum pressures (no greater than 1.06 times the maximum allowable working pressure (use of safety valves required)); loadings due to internal pressures (these set the minimum required thicknesses unless other loadings exceed 10% of the allowable working stress); thickness of cylindrical components under internal pressure as determined from formulae; openings and reinforcements; fatigue, fast-fracture, creep and other failure mechanisms; and hydrostatic proof tests (1.5 times the maximum allowable working pressure). Note, that Section III: Nuclear Power Plant Components uses the more sophisticated, but less historically based, design by analysis. The use of design by analysis in Section III is a precedent important for acceptance of CFCCs in the 'Code' since CFCCs may require recently developed reliability techniques employing computer algorithms for implementation in advanced designs.

Finally, Section I requires affixing a special 'Code' stamp and a proper nameplate to the components and resulting system, respectively, after complying with all the 'Code' requirements of design and construction. To document compliance, seven types of Manufacturers Data Report Forms must be completed.

Section II also has its roots in the original 1914 edition of the Code. The original materials specifications were developed in a joint effort of ASME and ASTM for ferrous and nonferrous materials. In addition, joint specifications with the American Welding Society (AWS) have since been developed for welding rods, electrodes and filler metals. Only those ASTM or AWS specifications required by ASME are addressed in Section II. The documentation of Section II is a four part compendium of materials data: Part A — Ferrous Materials Specifications; Part B — Nonferrous Material Specifications; Part C — Specification for Welding Rods, Electrodes, and Filler Metals; and Part D — Properties. Part D lists material properties for all materials accepted by Sections I, III, and VIII. Not only mechanical properties but also physical properties are contained in Part D. However, a major portion of the data contained in Part D are tables of stresses as functions of temperature.

Because of increasing interest in CFCCs from the power generation community along with similar continuing interest in impervious graphite from the chemical processing community, the Task Group on Graphite and Ceramic Pressure Equipment of the ASME Boiler and Pressure Vessel Code was formed in 1995. The task group was formed to address the possible explicit introduction of brittle materials (i.e. impervious graphite and ceramic composites) as acceptable, performance-based materials into the 'Code.' Subsequent division of the task group resulted in one of two smaller groups: Subtask Group on Ceramic Pressure Equipment, specifically targeted to CFCCs. Two possible paths were considered for the sub task group: (1) development of a 'Code'-case to demonstrate that brittle materials have been and are successfully being used in pressurized equipment and (2) development of a stand alone section of the 'Code.' Path (1) was chosen by the impervious graphite subtask group because there are many current applications of this material in pressure equipment. CMCs and CFCCs are not currently used in pressurized equipment; therefore, Path (2) was chosen. A 100+ page draft of a stand-alone section (cloned from Section X on PMCs) was subsequently developed with the following organization. Note that this draft section also employs reliability-based design rather than design by rule or factor of safety design by analysis.

1. Introduction.
2. General Requirements.

3. Material Requirements.
4. Design Requirements.
5. Fabrication Requirements.
6. Qualification Requirements.
7. Pressure Relief Devices.
8. Rules Governing Testing.
9. Inspection Requirements.
10. Marking, Stamping, Reporting.
11. Appendices.
12. Forms.

Work on this draft section is ongoing although tangible results are still in the future by several years.

3.2. Mil-Hdbk-17 CMC effort

Mil-Hdbk-17 is the outgrowth of a collaborative effort on the part of industry (i.e. defense contractors) and the US Department of Defense to clarify and codify issues involving the use of polymer matrix composites (PMCs) in advanced designs. Mil-Hdbk-17 has been in existence in various forms since 1959 and has been relatively successful in creating common language, design philosophies, fabrication methods, maintenance approaches, and certification of advanced PMCs. Recent directives from US Department of Defense have established the format for developing off-shoots of Mil-Hdbk-17 for other advanced composites, namely, metal matrix composites (MMCs) in 1993 and ceramic matrix composites (CMCs) in 1996. The organization of the Mil-Hdbk-17 effort for CMCs is shown in Fig. 11. The vision of the Mil-Hdbk-17 effort for CMCs is as follows:

> Mil-Hdbk-17 is the primary and authoritative source for characterization, statistically-based property and performance data of current and emerging ceramic matrix composites. It reflects the best available data and methodologies for characterization, testing, analysis and design, and usage guidelines in support of design methodologies for composites.

The objectives are:

- Development of a framework for the future successful use of CMCs.
- Provide guidance to industry for the collection of statistically meaningful critical data that designers need to utilize CMCs.
- Based on the requirements from the design community. identify appropriate properties and broadly accepted testing procedures — including consideration of the designation of the precision level and prioritization of properties required.
- Provide guidelines and recommendations for the characterization, testing, design, and utilization of CMC materials and structures.

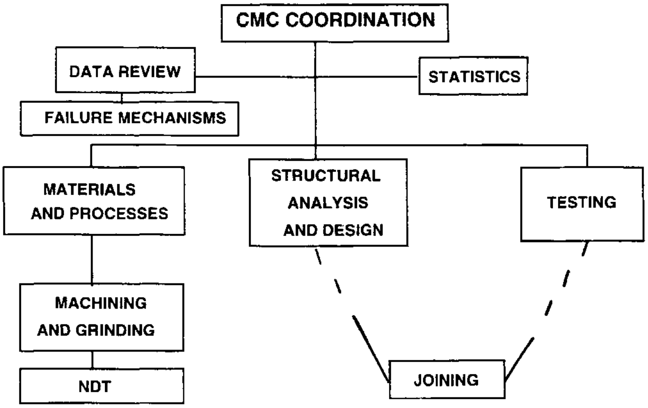


Figure 11. Organization of Mil-Hdbk-17 CMC effort.

- Provide the primary and authoritative sources for characterization, property, and performance data of current and emerging CMC systems.
- Provide recommendations for the statistical analysis of materials data and structures relativity.

Note that imbedded in the organization shown in Fig. 11 is an oversight activity for Data Review. Any data generated for inclusion in Mil-Hdbk-17 must satisfy requirements for confidence bounds and statistical sample size within a batch and batch to batch. Because of the need to establish the CMC portion of the Mil-Hdbk-17 as quickly as possible, data base generation has been simultaneous with establishing design rules and guidelines. Note also in Fig. 11 that while the Testing activity is separate, it can actually be thought of as performing a service role to the other activities. Moreover, the stated mission of the Testing activity is not to develop standards (this is best left to the standards writing bodies already discussed), but instead to identify and recommend those existing standards which are appropriate. Where appropriate standards do not exist, the Testing activity will assist standards writing bodies in the development of the standards.

Partly in support of the Mil-Hdbk-17 effort on CMCs, a multiple laboratory round-robin testing project was undertaken within a US Department of Energy program. Overall technical objectives of this project were as follows: (1) Procure 400 test specimens of a commercial CFCC. (2) Distribute these 400 test specimens to up to eight different laboratories for each type of test along with three current ASTM Test Methods: C1275-95 [17], C1292-95 [18], and C1341-95 [19], and the test parameters to complete testing. (3) Collect and analyze the results so as report the precision and bias for each of the standards using ASTM Practice E691 [20]. (4) Incorporate the results of the round-robin into a Mil-Hdbk-17 data base readily accessible to CFCC program participants for the purposes of designing, modeling and processing.

The material chosen for this study was a commercially-available CFCC (Sylramic 5200, Dow Corning Inc., Midland, Michigan, USA) with the following material description:

Fiber: Ceramic grade Nicalon™.

Fiber Form: 8HSW.

Interphase: Proprietary coating (containing boron nitride).

Lay-up: Warp alternated 0/90, Symmetric, 8 plies (0/90/0/90/90/0/90/0).

Matrix: SiNC (Sylramic™) via the polymer impregnation process (PIP).

Matrix filler: Silicon nitride.

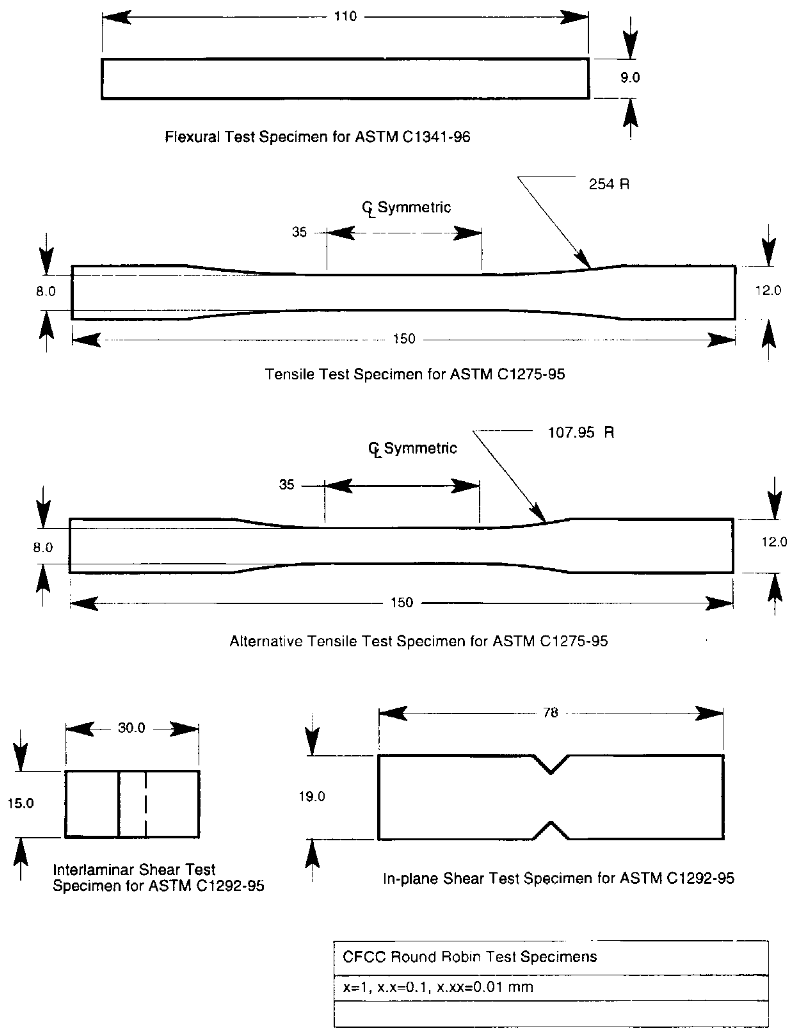


Figure 12. Round-robin test specimen for data base generation in support of Mil-Hdbk-17 CMC effort.

Using this lay-up eliminated the need to keep track of an overall warp direction. Test specimen shapes (see Fig. 12) were fabricated using conventional diamond grit cutting/grinding from plates of the material at an intermediate infiltration step, with processing continuing on the test specimen shapes up to the final infiltration. The 100 flexural and 100 tensile test specimens were cut such that the surface warp direction was along the length of the specimen. The 100 interlaminar and 100 in-plane shear specimens required notching (as illustrated by the dashed lines in Fig. 12) upon completion of the primary processing. Ten of each test specimen geometry were to be tested by up to 10 different academic, governmental, and industrial laboratories. At the time of this writing, the round robin was still ongoing, although all laboratory testing had been completed.

4. SUMMARY AND CONCLUSIONS

The main objective of this overview was to provide information on the current state of standards and codes for CFCCs which are a subset of CMCs. While the number and diversity of test standards for CFCCs is limited but growing, efforts are already underway to harmonize the various full-consensus industry/government standards through the international auspices of ISO. The success of the first ISO standard for CFCC will be instrumental in reducing redundancy and increasing productivity in development of CFCC standards. Future directions include developing standards for components, sub-components, environmental effects, electrical and electronic, thermo-mechanical, optical and biological characteristics.

Benefiting from the increasing number and diversity of test standards for CFCCs are design codes and data bases for these materials. Two current efforts — ASME Task Group on Boiler and Pressure Equipment and Mil-Hdbk-17 CMC effort — are helping to coalesce the range of ideas of design with and design of CFCCs. Various existing test methods are also being utilized to generate statistically significant data bases for inclusion within the design codes.

In conclusion, the development and implementation of standards and codes for CMCs is ongoing and rapidly progressing. As CMCs and in particular CFCCs move from materials to the development stage to prototype fabrication to final component production, not only standards but also the codes and attendant data will not only be essential but will also benefit from the maturation of this unique material system.

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REFERENCES

1. D. C. Phillips and R. W. Davidge, Test techniques for the mechanical properties of ceramic matrix fibre composites, *Brit. Ceram. Trans. J.* **85**, 123–130 (1986).
2. S. J. Schneider and D. R. Bradley, The standardization of advanced ceramics, *Adv. Ceram. Mater.* **3** (5), 442–449 (1988).
3. M. G. Jenkins and G. D. Quinn, ASTM Standards for Monolithic and Composite Advanced Ceramics: Industrial, Governmental and Academic Cooperation, paper 96-GT-270, American Society of Mechanical Engineers, New York (1996).
4. T. Kanno, International Standardization Activities for Fine Ceramics — Status of ISO/TC206 on Fine Ceramics, paper 96-GT-321, American Society of Mechanical Engineers, New York (1996).
5. M. Steen, European Standardization Efforts on Fibre-Reinforced Ceramic Matrix Composites, paper 96-GT-269, American Society of Mechanical Engineers, New York (1996).
6. C. Zweben, Ceramic matrix composite — Mechanical properties and test methods, *Ceram. Eng. Sci. Proc.* **6** (7–8), 499–503 (1985).
7. I. W. Donald and P. W. McMillan, Review: Ceramic matrix composites, *J. Mater. Sci.* **11**, 949–972 (1976).
8. M. G. Jenkins and M. D. Mello, Processing and characterization of 3-D braided, continuous SiC fibre reinforced/CVI matrix composites, in: *Processing, Design and Performance of Composite Materials*, MD-Vol. 52, pp. 239–254. American Society of Mechanical Engineers, New York, NY (1994).
9. M. G. Jenkins, J. P. Piccola, Jr. and E. Lara-Curzio, Onset of cumulative damage (first matrix cracking) and the effects of test parameters on the tensile behaviour of a continuous fibre-reinforced ceramic composite (CFCC), in: *Fracture Mechanics of Ceramics*, Vol. 12, R. C. Bradt, D. P. H. Hasselman, D. Munz, M. Sakai and V. Y. Shevchenko (Eds), pp. 267–282. Plenum Press, New York, NY (1996).
10. L. W. Rehfield, E. A. Armanios and Q. Changli, Analysis of behaviour of fibrous composite compression specimens, in: *Recent Advances in Composites in the United States and Japan, ASTM STP 864*, J. R. Vinson and M. Taya (Eds), pp. 236–252. ASTM, Philadelphia, PA (1985).
11. D. F. Adams, Mechanical testing of composite materials for quality control, in: *Third Int. Congress on Composites*, Weisbaden, Germany (1991).
12. T. R. Barnett and H. S. Starrett, Room and Elevated Temperature Mechanical and Thermal Properties of Corning Nicalon CAS, WRDC-TR-90-4131 (1992).
13. D. C. Cranmer, Workshop on assessment of testing methodology for glass, glass-ceramic, and ceramic matrix composites, *J. Resch. NIST* **96** (4), 493–501 (1991).
14. C. X. Campbell, Thermomechanical Response of a Continuous Fiber Ceramic Composite (CFCC) Resulting from Oxidation, Temperature Degradation, and Residual Stress, Master of Science Thesis, University of Washington, Seattle, Washington (1998).
15. American Society of Mechanical Engineers, ASME Boiler and Pressure Vessel Code, ASME, New York (1983).
16. J. R. Farr, The ASME Boiler and Pressure Vessel Code: Overview, in: *Pressure Vessel Codes and Standards*, pp. 1–34. Elsevier Applied Science (1983).
17. ASTM, C1275-94, Standard Test Method for Monotonic Tensile Strength Testing of Continuous Fibre-Reinforced Advanced Ceramics with Solid Rectangular Cross-Section Specimens at Ambient Temperatures, Annual Book of ASTM Standards, Vol. 15.01, American Society for Testing and Materials, W. Conshohocken, PA, USA (1998).
18. ASTM, C1292-96, Standard Test Method for Shear Strength of Continuous Fibre-Reinforced Advanced Ceramics at Ambient Temperatures, Annual Book of ASTM Standards, Vol. 15.01, American Society for Testing and Materials, W. Conshohocken, PA, USA (1998).

19. ASTM, C1341-96, Standard Test Method for Flexural Properties of Continuous Fiber-Reinforced Advanced Ceramics, Annual Book of ASTM Standards, Vol. 15.01, American Society for Testing and Materials, W. Conshohocken, PA, USA (1998).
20. ASTM, E691-92, Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method, Annual Book of ASTM Standards, American Society for Testing and Materials, W. Conshohocken, PA, USA (1998).