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**DEPARTMENT OF DEFENSE  
HANDBOOK**

**COMPOSITE MATERIALS HANDBOOK**

**VOLUME 5. CERAMIC MATRIX COMPOSITES**



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3. Every effort has been made to reflect the latest information on polymer (organic), metal, and ceramic composites. The handbook is continually reviewed and revised to ensure its completeness and currentness. Documentation for the secretariat should be directed to: Materials Sciences Corporation, MIL-HDBK-17 Secretariat, 500 Office Center Drive, Suite 250, Fort Washington, PA 19034..
4. MIL-HDBK-17 provides guidelines and material properties for polymer (organic), metal, and ceramic matrix composite materials. The first three volumes of this handbook currently focus on, but are not limited to, polymeric composites intended for aircraft and aerospace vehicles. Metal matrix composites (MMC) and ceramic matrix composites (CMC), including carbon-carbon composites (C-C) are covered in Volume 4 and Volume 5 , respectively.
5. This standardization handbook has been developed and is being maintained as a joint effort of the Department of Defense and the Federal Aviation Administration.
6. The information contained in this handbook was obtained from materials producers, industry, reports on Government sponsored research, the open literature, and by contact with research laboratories and those who participate in the MIL-HDBK-17 coordination activity.
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**DEPARTMENT OF DEFENSE  
HANDBOOK**

**CERAMIC MATRIX COMPOSITES**

**VOLUME 5**

**PART A. INTRODUCTION AND GUIDELINES**

## **PART A. INTRODUCTION AND GUIDELINES**

### **1 MIL-17 GUIDELINES AND PROCEDURES**

This handbook documents engineering methodologies for the development of standardized, statistically-based material property data for ceramic matrix composite materials. Also provided are data summaries for a number of relevant composite material systems for which available data meet specific MIL-HDBK-17 requirements for publication. Additionally, supporting engineering and manufacturing technologies and common practices related to composite materials are summarized.

#### **1.1 INTRODUCTION**

It is generally understood that standardized, statistically-based, material property data are essential to an efficient engineering development process; such data are needed by material suppliers, engineering users, and system end-users alike. Since the inherent properties of materials are independent of specific applications, data development methodologies and material property data are applicable to a wide variety of industries. They also form much of the technical basis for establishment of statistically-based design values acceptable to procuring or certifying agencies. (An example of procuring agency is a branch of the U.S. Department of Defense, and an example of certifying agency is an office of the U.S. Federal Aviation Administration.) This evaluation of the inherent properties of ceramic matrix composite materials is the focus of MIL-HDBK-17 Volume 5. Material properties are continuously improving and those reported in this handbook are typical properties at the time of press.

##### **1.1.1 Objectives of Ceramic Matrix Composite (CMC) Working Groups**

Overall Vision for CMC:

MIL-HDBK-17 is the primary and authoritative source for characterization and statistically-based property and performance data of current and emerging advanced ceramic matrix composites. It reflects the best available data and methodologies for characterization, testing, analysis and design, and includes data development and usage guidelines in support of design methodologies for components.

Goals:

- A framework for the successful use of CMCs.
- Guidance to industry for the collection of statistically meaningful critical data that designers need to utilize CMCs.
- Appropriate prioritization of property requirements and broadly accepted testing procedures - including a consideration of the designation of the precision level, based on the requirements from the design community.
- Guidelines and recommendations for the characterization, processing, testing, design, and utilization of ceramic matrix composite materials and structures.
- The primary and authoritative source for characterization, property, and performance data of current and emerging ceramic matrix composite systems.
- Recommendations for the statistical analysis of materials data and structures reliability.

Objectives:

- 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.



## Volume 5, Part A Introduction and Guidelines

- Based on the requirements from the design community, identify appropriate properties and broadly accepted testing procedures - including a 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 ceramic matrix composite materials and structures.
- Provide the primary and authoritative source for characterization, property, and performance data of current and emerging ceramic matrix composite systems.
- Provide recommendations for the statistical analysis of materials data and structures reliability.

*1.1.1.1 Objectives and tasks for Data Review Working Group*

- Develop a framework for the future successful use of CMCs
- Guide the industrial base to collect statistically meaningful critical data that designers need to utilize CMCs
- Identify and prioritize appropriate properties and testing procedures
- Establish levels of precision required based on requirements for the design community
- Provide a methodology for the determination of key material characteristics (composition, microstructure and defects) critical for the performance of the material, perhaps including processing information
- Collect representative sets of data using the aforesaid methodologies on selected families of CMCs, proceed from materials with significant availability of data to those with minimal data but high potential.

*1.1.1.2 Vision, goals and objectives for Materials and Processes Working Group*

## Vision:

- To be the primary and authoritative source for information on the composition, fabrication, and characterization of CMC engineering materials and structures.

## Goals:

- To define the essential elements of information on composition, structure, and processing of CMCs necessary to design, select, fabricate, and utilize CMC structures.
- To specify the methods and procedures to be used in the characterization of ceramic matrix composites and their constituents.
- To provide a comprehensive overview of ceramic matrix composite technology, outlining the history, applications, benefits, ceramic composite systems, methods of fabrication, quality control, and supportability.

## Objectives:

- Define the materials and processing data elements and formats for the CMC data sheets.
- Define materials and processing data package requirements, in terms of nomenclatures, methods, formats and priorities.
- Provide detailed guidance on methods and procedures for characterizing CMC composites and their constituents.
- Prepare the Introduction section (history, applications, benefits, CMC systems, etc.) of the CMC handbook.
- Prepare the Materials, Processing, & Fabrication section of the CMC handbook. Prepare the Supportability section of the CMC handbook.

*1.1.1.3 Vision, goals and objectives for Structural Analysis & Design Codes Working Group*

## Vision:

- To be the primary and authoritative source for information and methodologies needed for the design and analysis of CMC engineering materials and structures.

## Goals:

- To define the information and methodologies needed for the design and analysis of engineering structures with CMCs
- To specify material property and performance input and validation data needed for design analysis
- To identify test parameters to produce those data, and specify analysis methodologies necessary to interpret such data, to ensure compatibility with design needs

## Objectives:

- Define inputs needed for the design analysis of CMCs
- Identify approaches and methodologies for the design and analysis of CMC materials and structures
- Identify approaches and methodologies for the design and analysis of CMC structural joints

*1.1.1.4 Vision, goals and objectives for Testing Working Group*

## Vision:

- To be the primary and authoritative source for recommended/required methods for testing and characterization of CMCs and their constituent materials.

## Goals:

- To identify appropriate existing consensus standard test methods for CMCs and their constituent materials.
- To assist in the development of appropriate standard test methods for CMCs and their constituent materials, where no such standards exist.

## Objectives:

- Incorporate appropriate existing standards for CMCs and their constituents in the Handbook. Incorporate appropriate new standards/draft standards for CMCs and their constituents in the Handbook.

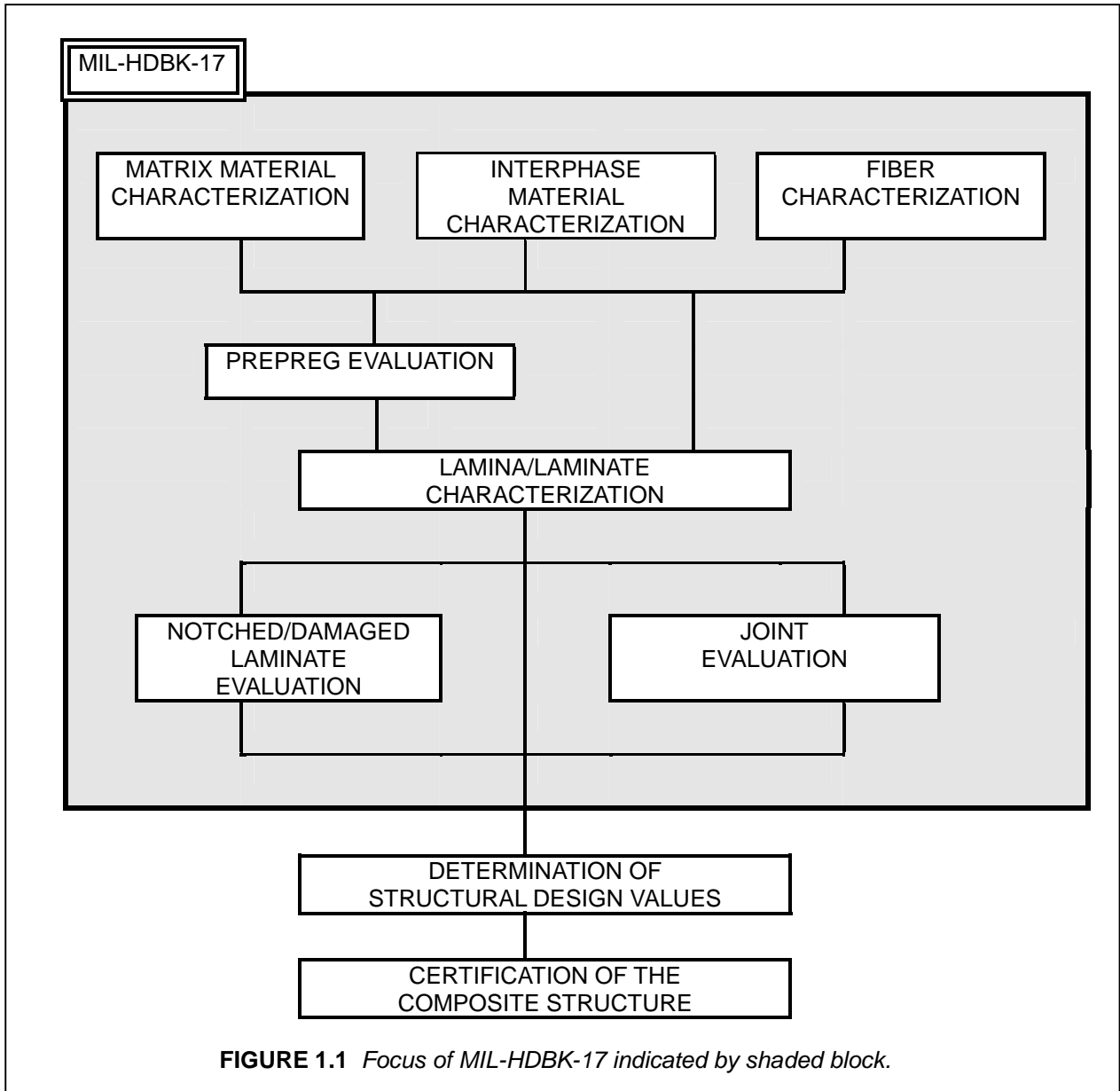
**1.2 PURPOSE**

The primary purpose of MIL-HDBK-17 is the standardization of engineering data development methodologies related to characterization testing, data reduction, and data reporting of properties for ceramic matrix composite materials. In support of this objective MIL-HDBK-17 publishes properties on composite material systems for which data meeting specific requirements are available. In addition, MIL-HDBK-17 provides selected guidance on other technical topics related to composites, including material selection, material specification, material processing, design, analysis, quality control, and repair of typical ceramic matrix composite materials. Thus, MIL-HDBK-17, Volume 5, is one volume with four parts, and serves as a source for the following:

- *Part A:* Documents material characterization data development methodology guidelines adaptable to a wide variety of needs, as well as specific requirements to be met by data published in the handbook. Most procuring and certifying agencies prefer, and some may require, that com-

posite material systems used in critical applications either be characterized in accordance with Part A guidelines or selected from material systems published in Part D.

- *Part B*: Serves as a source for additional technical guidance and lessons learned on a wide variety of disciplines related to ceramic matrix composites.
- *Part C*: Provides technical guidance on the test methodologies and statistical assessment of material characterization.
- *Part D*: Provides a repository of potential design data. The documented property summaries for material systems provide data meeting the criteria for any of the three MIL-HDBK-17 data documentation classes, (screening, interim, and fully approved).



**FIGURE 1.1** Focus of MIL-HDBK-17 indicated by shaded block.

## 1.3 SCOPE

For Department of Defense purposes, this handbook is for guidance only. This handbook cannot be cited as a requirement. If it is, the contractor does not have to comply. This mandate is a DoD requirement only; it is not applicable to the Federal Aviation Administration (FAA) or other government agencies.

The four parts of MIL-HDBK-17, Volume 5, serve as a general Reference source for technical information on ceramic matrix composites, including:

### 1.3.1 Part A: Introduction and Guidelines

This part contains guidelines for determining the properties of composite material systems, their constituents, and generic structural elements, including test planning, test matrices, sampling, conditioning, test procedure selection, data reporting, data reduction, statistical analysis, and other related topics. Part A contains *guidelines* for general development of material characterization data as well as *specific requirements* for publication of material data in MIL-HDBK-17.

It must be emphasized that this handbook differentiates between material basis values (material allowables) and design allowable values. Material basis values, being an intrinsic property of a composite material system, are the focus of this handbook. Design allowable values, while often rooted in material basis values, are application dependent, and include specific additional considerations that may further affect the strength or stiffness of the structure. Also, when establishing application design values there may be additional certification or procurement agency requirements that go beyond MIL-HDBK-17.

### 1.3.2 Part B: Design Supportability

Part B provides methodologies and lessons learned for the design, manufacture, analysis, and supportability of composite structures, and for utilization of the material data provided in Part D consistent with the guidance provided in Part A. Topics discussed in Part B include materials and processing, quality control, design and analysis, joints, reliability, thick composites, and supportability.

### 1.3.3 Part C: Testing

Part C addresses issues related to test methods and requirements for submission of data to MIL-17.

### 1.3.4 Part D: Data Requirements and Data Sets

Part D contains statistically-based data meeting specific MIL-HDBK-17 population sampling and data documentation requirements, covering constituents and material systems of general interest. Data published in Part D are under the jurisdiction of the Data Review Working Group and are approved by the overall Coordination Group (The MIL-HDBK-17 Coordination Group and Working Groups are discussed in Section 1.1). New material systems will be included and additional material data for existing systems will be added as approved data becomes available.

The material properties in Part D are defined over a range of potential use conditions, focusing, when possible, on the upper and lower material environmental limits so that application-specific environments do not limit use of the data. Special attention is given to the statistical treatment and analysis of data. Data at intermediate environmental conditions, when available, provide additional definition of the relation between material response and environment.

While the process of establishing structural design values for specific applications can begin with the data contained in Part D, most applications require collection of additional data, especially if there are requirements for data from the laminate or higher structural complexity levels. Also, the ability to manufacture material equivalent to that from which the data in Part D were obtained typically must be proven to the procuring or certifying agency, which usually involves limited testing and data comparison. General

guidelines for such material/process equivalence evaluation are presented in Part A; however, many of the details of such an evaluation remain at the discretion of the procuring or certifying agency.

## 1.4 USE OF THE DOCUMENT AND LIMITATIONS

### 1.4.1 Source of information

The information contained in MIL-HDBK-17 is obtained from materials producers and fabricators, the aerospace industry, reports on government-sponsored research, open literature, direct contacts with researchers, and from participants in MIL-HDBK-17 coordination activities. All information published in this document has been coordinated and reviewed by representatives from industry, the US Army, the US Air Force, US Navy, the US Federal Aviation Administration, the Department of Energy, and NASA. Every effort has been made to reflect the most up-to-date information on the use of composite materials, with particular emphasis on use of composites in structures. The handbook is continually reviewed and revised to keep current with the state-of-the-art in order to ensure completeness and accuracy.

### 1.4.2 Use of data and guidelines in applications

All data contained herein are based on small-scale test specimens, predominantly loaded quasi-statically and uniaxially, for specific environmental conditions.<sup>1</sup> It is the user's responsibility to determine if handbook data are appropriate for a given application and to translate or scale the data as necessary for use:

- in a multi-directional laminate,
- on a structure of different characteristic size and geometry,
- under a multi-directional stress state,
- when exposed to a different environment, and/or
- when subjected to non-static loading.

Further discussions of these and other issues are provided in Part D. Specific uses of handbook data are beyond the scope and responsibility of MIL-HDBK-17, and applicability and interpretation of specific provisions of this handbook may require approval by an appropriate procurement or certification agency.

### 1.4.3 Strength properties and allowables terminology

The handbook intent is to provide guidelines for generating material property data, including statistically-based strength data at environmental extremes that bracket most intermediate application-specific environments. The philosophy is to avoid having application-specific issues govern generic material property characterization programs. If data are also available at intermediate environmental conditions, they can be used to more completely define the relationship between the property and the effect of the environment on that property. However, in some cases an environmental limit for a composite material system may be application dependent, and in others, data at environmental limits may not be available.

Available statistically-based strength data are tabulated in Part D. These data are useful as a starting point for establishing structural design allowable values when stress and strength analysis capabilities permit lamina-level margin-of-safety calculations. For such cases the MIL-HDBK-17 strength basis value may also be termed a material design allowable. Depending on the application, some structural design allowables may have to be empirically determined from additional laminate, element, or higher-level test data not provided by MIL-HDBK-17.

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<sup>1</sup>Unless otherwise noted, tests were conducted in conformance with the particular test method noted. The emphasis is on data obtained from ASTM standard test methods for advanced composites, but where an ASTM test method has been deemed inappropriate or is not yet available, or when data from a nonstandard but commonly practiced test procedure are available, then data from a non-standard test method may have been accepted for publication. The specific test method used is noted in the data documentation. See also the statement on test method acceptance criteria in Volume 1, Section 2.5.5.

#### 1.4.4 Use of References

While many References are provided at the end of each chapter, note that the information in these citations may not necessarily comply in every respect either with the general guidelines for data development or with the specific requirements for publication of data in the handbook. The References are simply intended to be helpful, but not necessarily complete or authoritative sources of additional related information on specific subject areas.

#### 1.4.5 Use of tradenames and product names

Use of tradenames or proprietary product names does *not* constitute an endorsement of those products by the US Government or by the MIL-HDBK-17 Coordination Group.

#### 1.4.6 Toxicity, health hazards, and safety

Certain processing and test methods discussed in MIL-HDBK-17 may involve hazardous materials, operations, or equipment. These methods may not address safety problems, if any, associated with their use. It is the responsibility of the user of these methods to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use. The user is referred to the Advanced Composite Materials US Army Interim Health and Safety Guidance for a discussion of the health and safety issues involved in the processing and use of composite materials. This document is generated by the US Army Environmental Hygiene Agency, Aberdeen Proving Ground, MD. Material manufacturers, as well as various composites user groups, may also provide guidance on health and safety issues pertinent to composite materials.

#### 1.4.7 Ozone depleting chemicals

Restrictions on the use of ozone depleting chemicals are detailed in the US Clean Air Act of 1991.

### 1.5 APPROVAL PROCEDURES

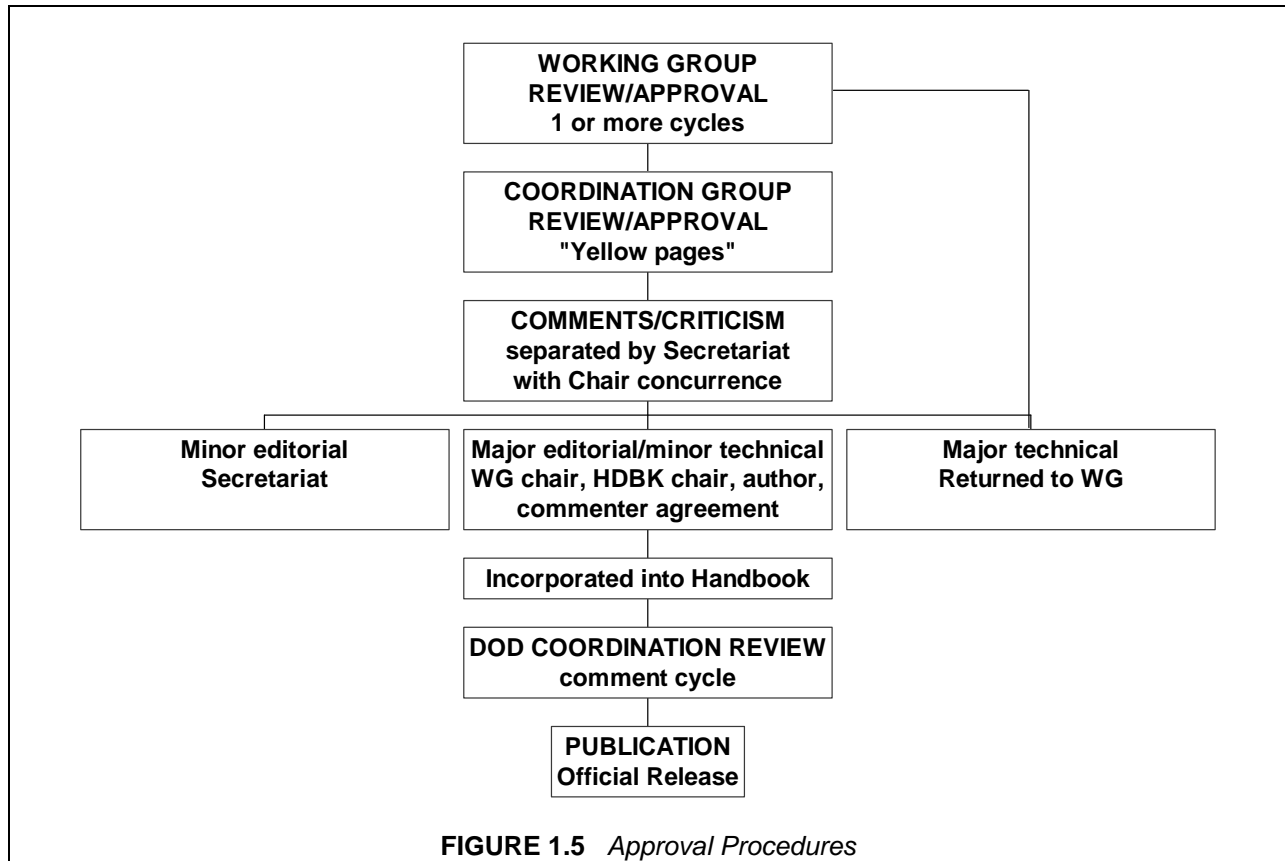
The content of the handbook is developed and approved by the MIL-HDBK-17 Coordination Group as shown in Figure 1.5, which meets every eight months to consider changes and additions to the handbook. This Group consists of the handbook Co-Chairs, Coordinator, Secretariat, Working Group Chairs, and the active Working Group participants, which include representatives from various US and international procuring and certifying agencies, in addition to the producing industries and academic and research institutions. MIL-HDBK-17 Coordination Group meetings are announced to participants by mail about eight weeks prior to the scheduled meeting date, and minutes of the meetings are mailed eight weeks following the close of the meeting.

While each of the Working Groups functions similarly, they are of two types: *Executive*, a single Working Group with oversight responsibility composed of the Working Group Chairs, the handbook Co-Chairs, Coordinator, and Secretariat; and *Standing*, including Data Review, Materials and Processing, Structural Analysis and Design, and Testing Working Groups. The makeup and organization of the Coordination Group and Working Groups, as well as the procedures followed for document change approval, are summarized in the MIL-HDBK-17 Coordination Group Member's Guide, separately published and available from either the Coordinator or Secretariat.

Proposals for addition to, deletion from, or modification to the handbook should be submitted to both the appropriate Working Group and the Secretariat well in advance of the announcement mailing date, and should include specific notation of the proposed changes and adequate documentation of supporting data or analytical procedures. Reproducible copies of figures, drawings, or photographs proposed for publication in the document should be furnished to the Secretariat. Following approval by the appropriate Working Group, the proposed changes are published in the next minutes of the Coordination Group, in a

special section of the minutes called the "yellow pages", and all participants are allowed comment on the proposed changes. If no substantive comments are received on any individual item by the posted response date, then that item is considered approved by the Coordination Group and is considered effective as of that date. (Prior to publication in the next revision of the handbook the collected changes are reviewed by various branches of the US DoD. Additional proposals for revision may result from this US DoD review.)

Requests for inclusion of material property data into MIL-HDBK-17 should be submitted to either the Coordinator or the Secretariat, accompanied by the documentation specified in Part D, Chapter 17. A Data Source Information Package has been created to aid those considering submitting data for inclusion in MIL-HDBK-17, and is available from either the Coordinator or the Secretariat. The Secretariat reviews and analyzes each data submission and, at the next available meeting of the Coordination Group, presents a summary for evaluation by the Data Review Working Group. The choice of new materials to be included herein is governed by the MIL-HDBK-17 Coordination Group. Practical considerations preclude inclusion of all advanced composite materials, but reasonable attempts will be made to add new material systems of interest in a timely manner.



## 1.6 SYMBOLS, ABBREVIATIONS, AND SYSTEMS OF UNITS

This section defines the symbols and abbreviations which are used within MIL-HDBK-17 and describes the system of units which is maintained. Common usage is maintained where possible. References 1.6(a), 1.6(b), and 1.6(c) served as primary sources for this information.

### 1.6.1 Symbols and abbreviations

The symbols and abbreviations used in this document are defined in this section with the exception of statistical symbols. These latter symbols are defined in Part C, Section 16.1.3. The lamina/laminate coordinate axes used for all properties and a summary of the mechanical property notation are shown in Figure 1.6.1.

- The symbols  $f$  and  $m$ , when used as either subscripts or superscripts, always denote fiber and matrix, respectively.
- The type of stress (for example,  $c_y$  - compressive yield) is always used in the superscript position.
- Direction indicators (for example,  $x, y, z, 1, 2, 3$ , etc.) are always used in the subscript position.
- Ordinal indicators of laminae sequence (e.g., 1, 2, 3, etc.) are used in the superscript position and must be parenthesized to distinguish them from mathematical exponents.
- Other indicators may be used in either subscript or superscript position, as appropriate for clarity.
- Compound symbols (such as, basic symbols plus indicators) which deviate from these rules are shown in their specific form in the following list.

The following general symbols and abbreviations are considered standard for use in MIL-HDBK-17. Where exceptions are made, they are noted in the text and tables.

A	-	(1) area ( $m^2, in^2$ )
	-	(2) ratio of alternating stress to mean stress
	-	(3) A-basis for mechanical property values
a	-	(1) length dimension (mm, in)
	-	(2) acceleration ( $m/sec^2, ft/sec^2$ )
	-	(3) amplitude
	-	(4) crack or flaw dimension (mm, in)
B	-	(1) B-basis for mechanical property values
	-	(2) biaxial ratio
Btu	-	British thermal unit(s)
b	-	width dimension (mm, in), e.g., the width of a bearing or compression panel normal to load, or breadth of beam cross-section
C	-	(1) specific heat ( $kJ/kg$ °C, $Btu/lb$ °F)
	-	(2) Celsius
CF	-	centrifugal force (N, lbf)
CPF	-	crossply factor
CPT	-	cured ply thickness (mm, in.)
CG	-	(1) center of mass, "center of gravity"
	-	(2) area or volume centroid
$\mathcal{C}$	-	centerline
$c$	-	column buckling end-fixity coefficient
$\bar{c}$	-	honeycomb sandwich core depth (mm, in)
cpm	-	cycles per minute
D	-	(1) diameter (mm, in)
	-	(2) hole or fastener diameter (mm, in)
	-	(3) plate stiffness (N-m, lbf-in)
d	-	mathematical operator denoting differential



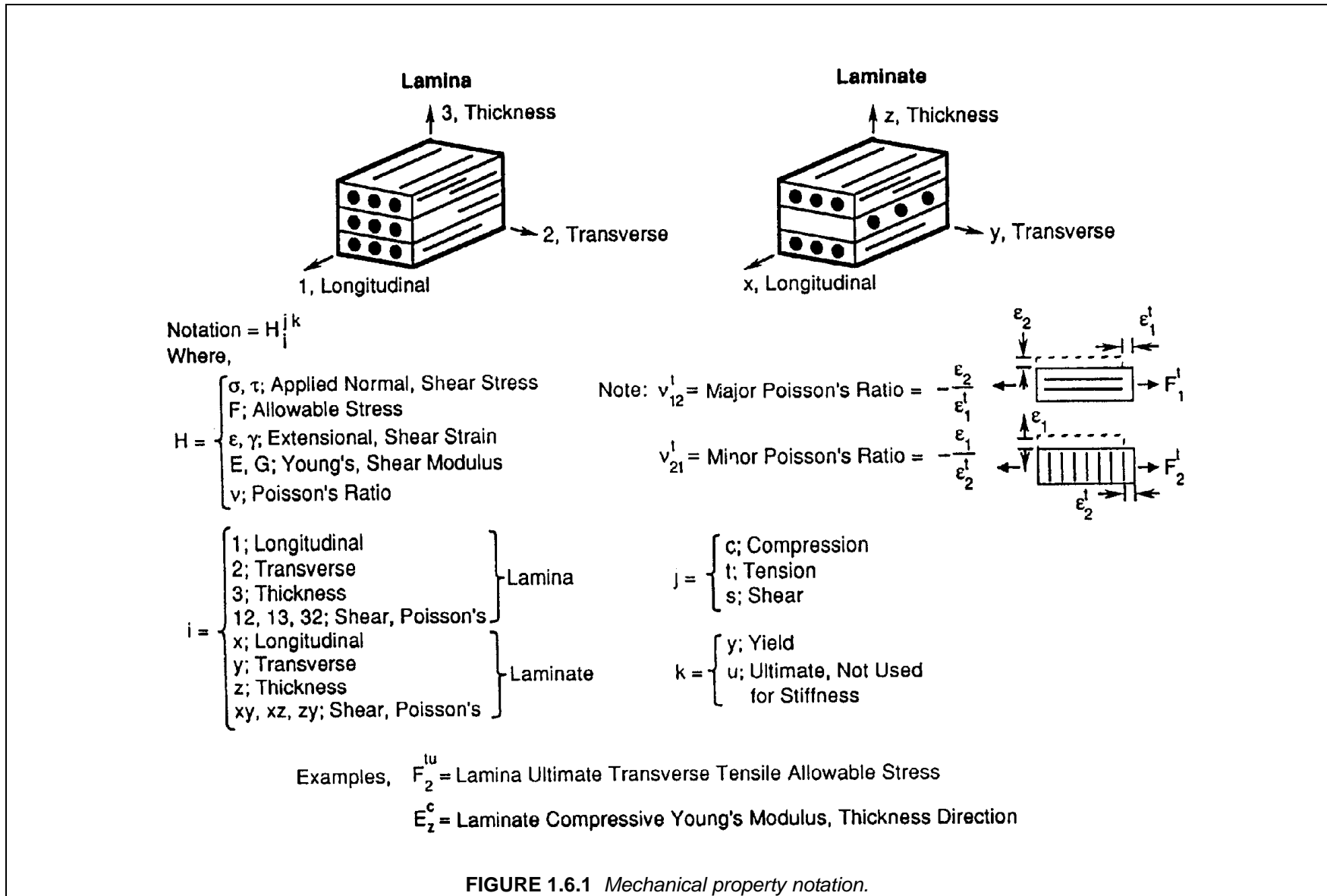


FIGURE 1.6.1 Mechanical property notation.

E	-	modulus of elasticity in tension, average ratio of stress to strain for stress below proportional limit (GPa,Msi)
E'	-	storage modulus (GPa,Msi)
E''	-	loss modulus (GPa,Msi)
E <sub>c</sub>	-	modulus of elasticity in compression, average ratio of stress to strain for stress below proportional limit (GPa,Msi)
E <sub>c</sub> '	-	modulus of elasticity of honeycomb core normal to sandwich plane (GPa,Msi)
E <sup>sec</sup>	-	secant modulus (GPa,Msi)
E <sup>tan</sup>	-	tangent modulus (GPa,Msi)
e	-	minimum distance from a hole center to the edge of the sheet (mm,in)
e/D	-	ratio of edge distance to hole diameter (bearing strength)
F	-	(1) stress (MPa,ksi)
	-	(2) Fahrenheit
F <sup>b</sup>	-	bending stress (MPa,ksi)
F <sup>ccr</sup>	-	crushing or crippling stress (upper limit of column stress for failure) (MPa,ksi)
F <sup>su</sup>	-	ultimate stress in pure shear (this value represents the average shear stress over the cross-section) (MPa,ksi)
FAW	-	fiber areal weight (g/m <sup>2</sup> , lb/in <sup>2</sup> )
FV	-	fiber volume (%)
f	-	(1) internal (or calculated) stress (MPa,ksi)
	-	(2) stress applied to the gross flawed section (MPa,ksi)
	-	(3) creep stress (MPa,ksi)
f <sup>c</sup>	-	internal (or calculated) compressive stress (MPa,ksi)
f <sub>c</sub>	-	(1) maximum stress at fracture (MPa,ksi)
	-	(2) gross stress limit (for screening elastic fracture data (MPa,ksi)
ft	-	foot, feet
G	-	modulus of rigidity (shear modulus) (GPa,Msi)
GPa	-	gigapascal(s)
g	-	(1) gram(s)
	-	(2) acceleration due to gravity (m/s <sup>2</sup> ,ft/s <sup>2</sup> )
H/C	-	honeycomb (sandwich)
h	-	height dimension (mm,in) e.g., the height of a beam cross-section
hr	-	hour(s)
I	-	area moment of inertia (mm <sup>4</sup> ,in <sup>4</sup> )
i	-	slope (due to bending) of neutral plane in a beam, in radians
in.	-	inch(es)
J	-	(1) torsion constant (= I <sub>p</sub> for round tubes) (m <sup>4</sup> ,in <sup>4</sup> )
	-	(2) Joule
K	-	(1) Kelvin
	-	(2) stress intensity factor (MPa/m,ksi/in)
	-	(3) coefficient of thermal conductivity (W/m °C, Btu/ft <sup>2</sup> /hr/in/°F)
	-	(4) correction factor
	-	(5) dielectric constant
K <sub>app</sub>	-	apparent plane strain fracture toughness or residual strength (MPa/m,ksi/in)
K <sub>c</sub>	-	critical plane strain fracture toughness, a measure of fracture toughness at point of crack growth instability (MPa/m,ksi/in)
K <sub>lc</sub>	-	plane strain fracture toughness (MPa/m,ksi/in)
K <sub>N</sub>	-	empirically calculated fatigue notch factor
K <sub>s</sub>	-	plate or cylinder shear buckling coefficient
K <sub>t</sub>	-	(1) theoretical elastic stress concentration factor
	-	(2) t <sub>w</sub> /c ratio in H/C sandwich
K <sub>v</sub>	-	dielectric strength (KV/mm, V/mil)
K <sub>x</sub> ,K <sub>y</sub>	-	plate or cylinder compressive buckling coefficient
k	-	strain at unit stress (m/m,in/in)

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L	-	cylinder, beam, or column length (mm,in)
L'	-	effective column length (mm,in)
lb	-	pound
M	-	applied moment or couple (N-m,in-lbf)
Mg	-	megagram(s)
MPa	-	megapascal(s)
m	-	(1) mass (kg,lb)
	-	(2) number of half wave lengths
	-	(3) metre
	-	(4) slope
N	-	(1) number of fatigue cycles to failure
	-	(2) number of laminae in a laminate
	-	(3) distributed in-plane forces on a panel (lbf/in)
	-	(4) Newton
	-	(5) normalized
NA	-	neutral axis
n	-	(1) number of times in a set
	-	(2) number of half or total wavelengths
	-	(3) number of fatigue cycles endured
P	-	(1) applied load (N,lbf)
	-	(2) exposure parameter
	-	(3) probability
	-	(4) specific resistance ( $\Omega$ )
$P_f$	-	failure load
$P^u$	-	test ultimate load, (N,lb per fastener)
$P^y$	-	test yield load, (N,lb per fastener)
p	-	normal pressure (Pa,psi)
psi	-	pounds per square inch
Q	-	area static moment of a cross-section ( $\text{mm}^3, \text{in}^3$ )
q	-	shear flow (N/m,lbf/in)
R	-	(1) algebraic ratio of minimum load to maximum load in cyclic loading
	-	(2) reduced ratio
r	-	(1) radius (mm,in)
	-	(2) root radius (mm,in)
	-	(3) reduced ratio (regression analysis)
S	-	(1) shear force (N,lbf)
	-	(2) nominal stress in fatigue (MPa,ksi)
	-	(3) S-basis for mechanical property values
$S_a$	-	stress amplitude in fatigue (MPa,ksi)
$S_e$	-	fatigue limit (MPa,ksi)
$S_m$	-	mean stress in fatigue (MPa,ksi)
$S_{\max}$	-	highest algebraic value of stress in the stress cycle (MPa,ksi)
$S_{\min}$	-	lowest algebraic value of stress in the stress cycle (MPa,ksi)
$S_R$	-	algebraic difference between the minimum and maximum stresses in one cycle (MPa,ksi)
S.F.	-	safety factor
s	-	(1) arc length (mm,in)
	-	(2) H/C sandwich cell size (mm,in)
T	-	(1) temperature ( $^{\circ}\text{C}, ^{\circ}\text{F}$ )
	-	(2) applied torsional moment (N-m,in-lbf)
$T_d$	-	thermal decomposition temperature ( $^{\circ}\text{C}, ^{\circ}\text{F}$ )
$T_F$	-	exposure temperature ( $^{\circ}\text{C}, ^{\circ}\text{F}$ )
$T_g$	-	glass transition temperature ( $^{\circ}\text{C}, ^{\circ}\text{F}$ )
$T_m$	-	melting temperature ( $^{\circ}\text{C}, ^{\circ}\text{F}$ )

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t	- (1) thickness (mm,in)
	- (2) exposure time (s)
	- (3) elapsed time (s)
V	- (1) volume (mm <sup>3</sup> ,in <sup>3</sup> )
	- (2) shear force (N,lbf)
W	- (1) weight (N,lbf)
	- (2) width (mm,in)
	- (3) Watt
x	- distance along a coordinate axis
Y	- nondimensional factor relating component geometry and flaw size
y	- (1) deflection (due to bending) of elastic curve of a beam (mm,in)
	- (2) distance from neutral axis to given point
	- (3) distance along a coordinate axis
Z	- section modulus, I/y (mm <sup>3</sup> ,in <sup>3</sup> )
$\alpha$	- coefficient of thermal expansion (m/m/°C,in/in/°F)
$\gamma$	- shear strain (m/m,in/in)
$\Delta$	- difference (used as prefix to quantitative symbols)
$\delta$	- elongation or deflection (mm,in)
$\epsilon$	- strain (m/m,in/in)
$\epsilon^e$	- elastic strain (m/m,in/in)
$\epsilon^p$	- plastic strain (m/m,in/in)
$\mu$	- permeability
$\eta$	- plasticity reduction factor
[ $\eta$ ]	- intrinsic viscosity
$\eta^*$	- dynamic complex viscosity
$\nu$	- Poisson's ratio
$\rho$	- (1) density (kg/m <sup>3</sup> ,lb/in <sup>3</sup> )
	- (2) radius of gyration (mm,in)
$\rho_c$	- H/C sandwich core density (kg/m <sup>3</sup> ,lb/in <sup>3</sup> )
$\Sigma$	- total, summation
$\sigma$	- standard deviation
$\sigma_{ij}, \tau_{ij}$	- stress in j direction on surface whose outer normal is in i direction (i, j = 1, 2, 3 or x, y, z) (MPa,ksi)
T	- applied shear stress (MPa,ksi)
$\omega$	- angular velocity (radians/s)
$\infty$	- infinity

## 1.6.1.1 Constituent properties

The following symbols apply specifically to the constituent properties of a typical composite material.

$E^f$	- Young's modulus of filament material (MPa,ksi)
$E^m$	- Young's modulus of matrix material (MPa,ksi)
$E_{\bar{x}}^g$	- Young's modulus of impregnated glass scrim cloth in the filament direction or in the warp direction of a fabric (MPa,ksi)
$E_{\bar{y}}^g$	- Young's modulus of impregnated glass scrim cloth transverse to the filament direction or to the warp direction in a fabric (MPa,ksi)
$G^f$	- shear modulus of filament material (MPa,ksi)
$G^m$	- shear modulus of matrix (MPa,ksi)
$G_{\bar{x}y}^g$	- shear modulus of impregnated glass scrim cloth (MPa,ksi)
$G_{cx}^g$	- shear modulus of sandwich core along X-axis (MPa,ksi)

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$G_{cy}$	-	shear modulus of sandwich core along Y-axis (MPa,ksi)
$\ell$	-	filament length (mm,in)
$\alpha^f$	-	coefficient of thermal expansion for filament material (m/m/°C,in/in/°F)
$\alpha^m$	-	coefficient of thermal expansion for matrix material (m/m/°C,in/in/°F)
$\alpha_x^g$	-	coefficient of thermal expansion of impregnated glass scrim cloth in the filament direction or in the warp direction of a fabric (m/m/°C,in/in/°F)
$\alpha_y^g$	-	coefficient of thermal expansion of impregnated glass scrim cloth transverse to the filament direction or to the warp direction in a fabric (m/m/°C,in/in/°F)
$\nu^f$	-	Poisson's ratio of filament material
$\nu^m$	-	Poisson's ratio of matrix material
$\nu_{xy}^g$	-	glass scrim cloth Poisson's ratio relating to contraction in the transverse (or fill) direction as a result of extension in the longitudinal (or warp) direction
$\nu_{yx}^g$	-	glass scrim cloth Poisson's ratio relating to contraction in the longitudinal (or warp) direction as a result of extension in the transverse (or fill) direction
$\sigma$	-	applied axial stress at a point, as used in micromechanics analysis (MPa,ksi)
$\tau$	-	applied shear stress at a point, as used in micromechanics analysis (MPa,ksi)

## 1.6.1.2 Laminae and laminates

The following symbols, abbreviations, and notations apply to composite laminae and laminates. At the present time the focus in MIL-HDBK-17 is on laminae properties. However, commonly used nomenclature for both laminae and laminates are included here to avoid potential confusion.

$A_{ij}$ (i,j = 1,2,6)	-	extensional rigidities (N/m,lbf/in)
$B_{ij}$ (i,j = 1,2,6)	-	coupling matrix (N,lbf)
$C_{ij}$ (i,j = 1,2,6)	-	elements of stiffness matrix (Pa,psi)
$D_x, D_y$	-	flexural rigidities (N-m,lbf-in)
$D_{xy}$	-	twisting rigidity (N-m,lbf-in)
$D_{ij}$ (i,j = 1,2,6)	-	flexural rigidities (N-m,lbf-in)
$E_1$	-	Young's modulus of lamina parallel to filament or warp direction (GPa,Msi)
$E_2$	-	Young's modulus of lamina transverse to filament or warp direction (GPa,Msi)
$E_x$	-	Young's modulus of laminate along x Reference axis (GPa,Msi)
$E_y$	-	Young's modulus of laminate along y Reference axis (GPa,Msi)
$G_{12}$	-	shear modulus of lamina in 12 plane (GPa,Msi)
$G_{xy}$	-	shear modulus of laminate in xy Reference plane (GPa,Msi)
$h_i$	-	thickness of $i^{\text{th}}$ ply or lamina (mm,in)
$M_x, M_y, M_{xy}$	-	bending and twisting moment components (N-m/m, in-lbf/in in plate and shell analysis)
$n_f$	-	number of filaments per unit length per lamina
$Q_x, Q_y$	-	shear force parallel to z axis of sections of a plate perpendicular to x and y axes, respectively (N/m,lbf/in)
$Q_{ij}$ (i,j = 1,2,6)	-	reduced stiffness matrix (Pa,psi)
$u_x, u_y, u_z$	-	components of the displacement vector (mm,in)
$u_x^0, u_y^0, u_z^0$	-	components of the displacement vector at the laminate's midsurface (mm,in)
$V_v$	-	void content (% by volume)
$V_f$	-	filament content or fiber volume (% by volume)
$V_g$	-	glass scrim cloth content (% by volume)
$V_m$	-	matrix content (% by volume)
$V_x, V_y$	-	edge or support shear force (N/m,lbf/in)
$W_f$	-	filament content (% by weight)
$W_g$	-	glass scrim cloth content (% by weight)
$W_m$	-	matrix content (% by weight)

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$W_s$	- weight of laminate per unit surface area (N/m <sup>2</sup> , lbf/in <sup>2</sup> )
$\alpha_1$	- lamina coefficient of thermal expansion along 1 axis (m/m/°C, in/in/°F)
$\alpha_2$	- lamina coefficient of thermal expansion along 2 axis (m/m/°C, in/in/°F)
$\alpha_x$	- laminate coefficient of thermal expansion along general Reference x axis (m/m/°C, in/in/°F)
$\alpha_y$	- laminate coefficient of thermal expansion along general Reference y axis (m/m/°C, in/in/°F)
$\alpha_{xy}$	- laminate shear distortion coefficient of thermal expansion (m/m/°C, in/in/°F)
$\theta$	- angular orientation of a lamina in a laminate, i.e., angle between 1 and x axes (°)
$\lambda_{xy}$	- product of $\nu_{xy}$ and $\nu_{yx}$
$\nu_{12}$	- Poisson's ratio relating contraction in the 2 direction as a result of extension in the 1 direction <sup>1</sup>
$\nu_{21}$	- Poisson's ratio relating contraction in the 1 direction as a result of extension in the 2 direction <sup>1</sup>
$\nu_{xy}$	- Poisson's ratio relating contraction in the y direction as a result of extension in the x direction <sup>1</sup>
$\nu_{yx}$	- Poisson's ratio relating contraction in the x direction as a result of extension in the y direction <sup>1</sup>
$\rho_c$	- density of a single lamina (kg/m <sup>3</sup> , lb/in <sup>3</sup> )
$\bar{\rho}_c$	- density of a laminate (kg/m <sup>3</sup> , lb/in <sup>3</sup> )
$\phi$	- (1) general angular coordinate, (°) - (2) angle between x and load axes in off-axis loading (°)

## 1.6.1.3 Subscripts

The following subscript notations are considered standard in MIL-HDBK-17.

1, 2, 3	- laminae natural orthogonal coordinates (1 is filament or warp direction)
A	- axial
a	- (1) adhesive - (2) alternating
app	- apparent
byp	- bypass
c	- composite system, specific filament/matrix composition. Composite as a whole, contrasted to individual constituents. Also, sandwich core when used in conjunction with prime (') - (4) critical
cf	- centrifugal force
e	- fatigue or endurance
eff	- effective
eq	- equivalent
f	- filament
g	- glass scrim cloth
H	- hoop
i	- i <sup>th</sup> position in a sequence
L	- lateral
m	- (1) matrix - (2) mean
max	- maximum
min	- minimum
n	- (1) n <sup>th</sup> (last) position in a sequence

<sup>1</sup>The convention for Poisson's ratio should be checked before comparing different sources as different conventions are used.

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	-	(2) normal
p	-	polar
s	-	symmetric
st	-	stiffener
T	-	transverse
t	-	value of parameter at time t
x, y, z	-	general coordinate system
$\Sigma$	-	total, or summation
o	-	initial or Reference datum
( )	-	format for indicating specific, temperature associated with term in parentheses. RT - room temperature (21°C,70°F); all other temperatures in °F unless specified.

1.6.1.4 *Superscripts*

The following superscript notations are considered standard in MIL-HDBK-17.

b	-	bending
br	-	bearing
c	-	(1) compression
	-	(2) creep
cc	-	compressive crippling
cr	-	compressive buckling
e	-	elastic
f	-	filament
g	-	glass scrim cloth
is	-	interlaminar shear
(i)	-	i <sup>th</sup> ply or lamina
lim	-	limit, used to indicate limit loading
m	-	matrix
ohc	-	open hole compression
oht	-	open hole tension
p	-	plastic
pl	-	proportional limit
rup	-	rupture
s	-	shear
scr	-	shear buckling
sec	-	secant (modulus)
so	-	offset shear
T	-	temperature or thermal
t	-	tension
tan	-	tangent (modulus)
u	-	ultimate
y	-	yield
'	-	secondary (modulus), or denotes properties of H/C core when used with subscript c
CAI	-	compression after impact

1.6.1.5 *Acronyms*

The following acronyms are used in MIL-HDBK-17.

AA	-	atomic absorption
ACUT	-	air coupled ultrasonic testing system
AES	-	Auger electron spectroscopy
AIA	-	Aerospace Industries Association
ANL	-	Argonne National Laboratory
ANOVA	-	analysis of variance

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ARL	-	US Army Research Laboratory
ASNT	-	American Society of Nondestructive Testing
ASTM	-	American Society for Testing and Materials
BMI	-	bismaleimide
BVID	-	barely visible impact damage
CA	-	commercially available
CAI	-	compression after impact
CAT	-	computer aided tomography
CCA	-	composite cylinder assemblage
CFCC	-	continuous fiber ceramic composite
CLS	-	crack lap shear
CPT	-	cured ply thickness
CTA	-	cold temperature ambient
CTD	-	cold temperature dry
CTE	-	coefficient of thermal expansion
CV	-	coefficient of variation
CVD	-	chemical vapor deposition
CVI	-	chemical vapor infiltration
DCB	-	double cantilever beam
DDA	-	dynamic dielectric analysis
DLL	-	design limit load
DMA	-	dynamic mechanical analysis
DoD	-	Department of Defense
DoE	-	Department of Energy
DSC	-	differential scanning calorimetry
DT	-	dye penetrant testing
DTA	-	differential thermal analysis
ECT	-	eddy current testing
ENF	-	end notched flexure
EOL	-	end-of-life
ESCA	-	electron spectroscopy for chemical analysis
ESR	-	electron spin resonance
ETW	-	elevated temperature wet
FAA	-	Federal Aviation Administration
FAW	-	fiber aerial weight
FFF	-	field flow fractionation
FM	-	fibrous monolith
FMECA	-	Failure Modes Effects Criticality Analysis
FOD	-	foreign object damage
FTIR	-	Fourier transform infrared spectroscopy
FWC	-	finite width correction factor
GC	-	gas chromatography
GSCS	-	Generalized Self Consistent Scheme
HAW	-	hypersonic aerodynamic weapons
HDT	-	heat distortion temperature
HPLC	-	high performance liquid chromatography
ICP	-	inductively coupled plasma emission spectroscopy
ID	-	identification
IR	-	infrared
ISI	-	in-service inspection
ISS	-	ion scattering spectroscopy
JANNAF	-	Joint Army, Navy, NASA, and Air Force
LA	-	limited availability
LC	-	liquid chromatography
LCF	-	low cycle fatigue
LC	-	liquid chromatography



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LPT	-	laminate plate theory
LSS	-	laminate stacking sequence
MI	-	melt infiltration
MMB	-	mixed mode bending
MOL	-	material operational limit
MPIT	-	magnetic particle testing
MS	-	(1) mass spectroscopy
	-	(2) military standard
M.S.	-	margin of safety
MSDS	-	material safety data sheet
MTBF	-	Mean Time Between Failure
MW	-	molecular weight
MWD	-	molecular weight distribution
NAS	-	National Aerospace Standard
NASA	-	National Aeronautics and Space Administration
NDC	-	nondestructive characterization
NDE	-	nondestructive evaluation
NDI	-	nondestructive inspection
NDT	-	nondestructive testing
NMR	-	nuclear magnetic resonance
OSL	-	observed significance level
P	-	prototype
PC	-	pyrolytic carbon
PP	-	polymer pyrolysis
PEEK	-	polyether ether ketone
PT	-	dye penetrate testing
RA	-	reduction of area
RBSN	-	reaction bonded silicon nitride
RDS	-	rheological dynamic spectroscopy
RGT	-	radiographic testing
RH	-	relative humidity
RMS	-	root-mean-square
RT	-	room temperature
RTA	-	room temperature ambient
RTD	-	room temperature dry
RTM	-	resin transfer molding
RV	-	reentry vehicle
SACMA	-	Suppliers of Advanced Composite Materials Association
SBS	-	short beam shear strength
SAE	-	Society of Automotive Engineers
SANS	-	small-angle neutron scattering spectroscopy
SEC	-	size-exclusion chromatography
SEM	-	scanning electron microscopy
SFC	-	supercritical fluid chromatography
SI	-	International System of Units (Le Système International d'Unités)
SIMS	-	secondary ion mass spectroscopy
TBA	-	torsional braid analysis
TEM	-	transmission electron microscopy
TGA	-	thermogravimetric analysis
TLC	-	thin-layer chromatography
TMA	-	thermal mechanical analysis
TOS	-	thermal oxidative stability
TVM	-	transverse microcrack
UDC	-	unidirectional fiber composite
UT	-	ultrasonic testing
VNB	-	V-notched beam

WoF	-	work of fracture
XCT	-	X-ray computed tomography
XD	-	experimental development
XPS	-	X-ray photoelectron spectroscopy

### 1.6.2 System of units

To comply with Department of Defense Instructive 5000.2, Part 6, Section M, "Use of the Metric System," dated February 23, 1991, the data in MIL-HDBK-17 are generally presented in both the International System of Units (SI units) and the U. S. Customary (English) system of units. ASTM E-380, Standard for Metric Practice, provides guidance for the application for SI units which are intended as a basis for worldwide standardization of measurement units (Reference 1.6.2(a)). Further guidelines on the use of the SI system of units and conversion factors are contained in the following publications (References 1.6.2(b) - (e)):

- (1) DARCOM P 706-470, *Engineering Design Handbook: Metric Conversion Guide*, July 1976.
- (2) NBS Special Publication 330, "The International System of Units (SI)," National Bureau of Standards, 1986 edition.
- (3) NBS Letter Circular LC 1035, "Units and Systems of Weights and Measures, Their Origin, Development, and Present Status," National Bureau of Standards, November 1985.
- (4) NASA Special Publication 7012, "The International System of Units Physical Constants and Conversion Factors", 1964.

English to SI conversion factors pertinent to MIL-HDBK-17 data are contained in Table 1.6.2.

## 1.7 DEFINITIONS

The following definitions are used within MIL-HDBK-17. This glossary of terms is not totally comprehensive but it does represent nearly all commonly used terms. Where exceptions are made, they are noted in the text and Tables. For ease of identification the definitions have been organized alphabetically.

**A-Basis (or A-Value)** -- A statistically-based material property; a 95% lower confidence bound on the first percentile of a specified population of measurements. Also a 95% lower tolerance bound for the upper 99% of a specified population.

**A-Stage** -- An early stage in the reaction of thermosetting resins in which the material is still soluble in certain liquids and may be liquid or capable of becoming liquid upon heating. (Sometimes referred to as **resol**.)

**Absorption** -- A process in which one material (the absorbent) takes in or absorbs another (the absorbate).

**Accuracy** -- The degree of conformity of a measured or calculated value to some recognized standard or specified value. Accuracy involves the systematic error of an operation.

**Adhesion** -- The state in which two surfaces are held together at an interface by forces or interlocking action or both.

**Adhesive** -- A substance capable of holding two materials together by surface attachment. In the handbook, the term is used specifically to designate structural adhesives, those which produce attachments capable of transmitting significant structural loads.

**TABLE 1.6.2** *English to SI conversion factors.*

To convert from	to	Multiplied by
Btu (thermochemical)/in <sup>2</sup> -s	watt/meter <sup>2</sup> (W/m <sup>2</sup> )	1.634 246 E+06
Btu-in/(s-ft <sup>2</sup> -°F)	W/(m K)	5.192 204 E+02
degree Fahrenheit	degree Celsius (°C)	T = (T - 32)/1.8
degree Fahrenheit	kelvin (K)	T = (T + 459.67)/1.8
foot	meter (m)	3.048 000 E-01
ft <sup>2</sup>	m <sup>2</sup>	9.290 304 E-02
foot/second	meter/second (m/s)	3.048 000 E-01
ft/s <sup>2</sup>	m/s <sup>2</sup>	3.048 000 E-01
inch	meter (m)	2.540 000 E-02
in. <sup>2</sup>	meter <sup>2</sup> (m <sup>2</sup> )	6.451 600 E-04
in. <sup>3</sup>	m <sup>3</sup>	1.638 706 E-05
kilogram-force (kgf)	newton (N)	9.806 650 E+00
kgf/m <sup>2</sup>	pascal (Pa)	9.806 650 E+00
kip (1000 lbf)	newton (N)	4.448 222 E+03
ksi (kip/in <sup>2</sup> )	MPa	6.894 757 E+00
lbf-in	N-m	1.129 848 E-01
lbf-ft	N-m	1.355 818 E+00
lbf/in <sup>2</sup> (psi)	pascal (Pa)	6.894 757 E+03
lb/in <sup>2</sup>	gm/m <sup>2</sup>	7.030 696 E+05
lb/in <sup>3</sup>	kg/m <sup>3</sup>	2.767 990 E+04
Msi (10 <sup>6</sup> psi)	GPa	6.894 757 E+00
pound-force (lbf)	newton (N)	4.488 222 E+00
pound-mass (lb avoirdupois)	kilogram (kg)	4.535 924 E-01
torr	pascal (Pa)	1.333 22 E+02

\* The letter "E" following the conversion factor stands for exponent and the two digits after the letter "E" indicate the power of 10 by which the number is to be multiplied.

**ADK** -- Notation used for the k-sample Anderson-Darling statistic, which is used to test the hypothesis that k batches have the same distribution.

**Aliquot** -- A small, representative portion of a larger sample.

**Aging** -- The effect, on materials, of exposure to an environment for a period of time; the process of exposing materials to an environment for an interval of time.

**Ambient** -- The surrounding environmental conditions such as pressure or temperature.

**Anelasticity** -- A characteristic exhibited by certain materials in which strain is a function of both stress and time, such that, while no permanent deformations are involved, a finite time is required to establish equilibrium between stress and strain in both the loading and unloading directions.

**Angleply** -- Same as **Crossply**.

**Anisotropic** -- Not isotropic; having mechanical and/or physical properties which vary with direction relative to natural Reference axes inherent in the material.

**Areal Weight of Fiber** -- The weight of fiber per unit area of prepreg. This is often expressed as grams per square meter. See Table 1.6.2 for conversion factors.

**Artificial Weathering** -- Exposure to laboratory conditions which may be cyclic, involving changes in temperature, relative humidity, radiant energy and any other elements found in the atmosphere in various geographical areas.

**Aspect Ratio** -- In an essentially two-dimensional rectangular structure (e.g., a panel), the ratio of the long dimension to the short dimension. However, in compression loading, it is sometimes considered to be the ratio of the load direction dimension to the transverse dimension. Also, in fiber micro-mechanics, it is referred to as the ratio of length to diameter.

**Autoclave** -- A closed vessel for producing an environment of fluid pressure, with or without heat, to an enclosed object which is undergoing a chemical reaction or other operation.

**Autoclave Molding** -- A process similar to the pressure bag technique. The lay-up is covered by a pressure bag, and the entire assembly is placed in an autoclave capable of providing heat and pressure for curing the part. The pressure bag is normally vented to the outside.

**Axis of Braiding** -- The direction in which the braided form progresses.

**B-Basis (or B-Value)** -- A statistically-based material property; a 95% lower confidence bound on the tenth percentile of a specified population of measurements. Also a 95% lower tolerance bound for the upper 90% of a specified population. (See Volume 1, Section 8.1.4)

**B-Stage** -- An intermediate stage in the reaction of a thermosetting resin in which the material softens when heated and swells when in contact with certain liquids but does not entirely fuse or dissolve. Materials are usually precured to this stage to facilitate handling and processing prior to final cure. (Sometimes referred to as **resitol**.)

**Balanced Laminate** -- A composite laminate in which all laminae at angles other than 0 degrees and 90 degrees occur only in  $\pm$  pairs (not necessarily adjacent).

**Batch (or Lot)** -- For fibers and resins, a quantity of material formed during the same process and having identical characteristics throughout. For prepreps, laminae, and laminates, material made from one batch of fiber and one batch of resin.

**Bearing Area** -- The product of the pin diameter and the specimen thickness.

**Bearing Load** -- A compressive load on an interface.

**Bearing Yield Strength** -- The bearing stress at which a material exhibits a specified limiting deviation from the proportionality of bearing stress to bearing strain.

**Bend Test** -- A test of ductility by bending or folding, usually with steadily applied forces. In some instances the test may involve blows to a specimen having a cross section that is essentially uniform over a length several times as great as the largest dimension of the cross section.

**Bias** -The orientation that is oblique to the warp direction.

**Binder** -- A bonding resin used to hold strands together in a mat or preform during manufacture of a molded object.

**Binomial Random Variable** -- The number of successes in independent trials where the probability of success is the same for each trial.

**Birefringence** -- The difference between the two principal refractive indices (of a fiber) or the ratio between the retardation and thickness of a material at a given point.

**Bleeder Cloth** -- A nonstructural layer of material used in the manufacture of composite parts to allow the escape of excess gas and resin during cure. The bleeder cloth is removed after the curing process and is not part of the final composite.

**Bobbin** -- A cylinder or slightly tapered barrel, with or without flanges, for holding tows, rovings, or yarns.

**Bond** -- The adhesion of one surface to another, with or without the use of an adhesive as a bonding agent.

**Braid** -- A system of three or more yarns which are interwoven in such a way that no two yarns are twisted around each other.

**Braid Angle** -- The acute angle measured from the axis of braiding.

**Braid, Biaxial** -- Braided fabric with two-yarn systems, one running in the  $+\theta$  direction, the other in the  $-\theta$  direction as measured from the axis of braiding.

**Braid Count** -- The number of braiding yarn crossings per inch measured along the axis of a braided fabric.

**Braid, Diamond** -- Braided fabric with an over one, under one weave pattern, (1 x 1).

**Braid, Flat** -- A narrow bias woven tape wherein each yarn is continuous and is intertwined with every other yarn in the system without being intertwined with itself.

**Braid, Hercules** -- A braided fabric with an over three, under three weave pattern, (3 x 3).

**Braid, Jacquard** -- A braided design made with the aid of a jacquard machine, which is a shedding mechanism by means of which a large number of ends may be controlled independently and complicated patterns produced.

**Braid, Regular** -- A braided fabric with an over two, under two weave pattern (2 x 2).

**Braid, Square** -- A braided pattern in which the yarns are formed into a square pattern.

**Braid, Two-Dimensional** -- Braided fabric with no braiding yarns in the through thickness direction.

**Braid, Three-Dimensional** -- Braided fabric with one or more braiding yarns in the through thickness direction.

**Braid, Triaxial** -- A biaxial braided fabric with laid in yarns running in the axis of braiding.

**Braiding** -- A textile process where two or more strands, yarns or tapes are intertwined in the bias direction to form an integrated structure.

**Braze** -- To join using a filler material which melts at a lower temperature than either of the materials being joined.

**Broadgoods** -- A term loosely applied to prepreg material greater than about 12 inches in width, usually furnished by suppliers in continuous rolls. The term is currently used to designate both collimated uniaxial tape and woven fabric prepreps.

**Buckling (Composite)** -- A mode of structural response characterized by an out-of-plane material deflection due to compressive action on the structural element involved. In advanced composites, buckling may take the form not only of conventional general instability and local instability but also a micro-instability of individual fibers.

**Bundle** -- A general term for a collection of essentially parallel filaments or fibers.

**C-Stage** -- The final stage of the curing reaction of a thermosetting resin in which the material has become practically infusible and insoluble. (Normally considered fully cured and sometimes referred to as **resite**.)

**Capstan** -- A friction type take-up device which moves braided fabric away from the fell. The speed of which determines the braid angle.

**Carbon Fibers** -- Fibers produced by the pyrolysis of organic precursor fibers such as rayon, polyacrylonitrile (PAN), and pitch in an inert atmosphere. The term is often used interchangeably with "graphite"; however, carbon fibers and graphite fibers differ in the temperature at which the fibers are made and heat-treated, and the amount of carbon produced. Carbon fibers typically are carbonized at about 2400°F (1300°C) and assay at 93 to 95% carbon, while graphite fibers are graphitized at 3450 to 5450°F (1900 to 3000°C) and assay at more than 99% elemental carbon.

**Carrier** -- A mechanism for carrying a package of yarn through the braid weaving motion. A typical carrier consists of a bobbin spindle, a track follower, and a tensioning device.

**Caul Plates** -- Smooth metal plates, free of surface defects, the same size and shape as a composite lay-up, used immediately in contact with the lay-up during the curing process to transmit normal pressure and to provide a smooth surface on the finished laminate.

**Censoring** -- Data is right (left) censored at M, if, whenever an observation is less than or equal to M (greater than or equal to M), the actual value of the observation is recorded. If the observation exceeds (is less than) M, the observation is recorded as M.

**Ceramic Matrix Composite** -- A material consisting of two or more constituents where a ceramic matrix is normally the principal component and the additional constituents are incorporated to strengthen, toughen, and/or enhance the thermo-physical properties.

**Chain-Growth Polymerization** -- One of the two principal polymerization mechanisms. In chain-growth polymerization, the reactive groups are continuously regenerated during the growth process. Once started, the polymer molecule grows rapidly by a chain of reactions emanating from a particular reactive initiator which may be a free radical, cation or anion.

**Characterization** -- Describes those features of composition and structure (including defects) of a material that are significant for a particular preparation, study of properties, or use, and for reproduction of the material.

**Chemical Vapor Infiltration (CVI)** -- A fabrication process for CMCs based upon the infiltration of gaseous reactants into a fiber/whisker preform leading to chemical reactions and the formation/deposition of a solid matrix.

**Chemical Vapor Deposition (CVD)** -- A process for depositing a solid material on a surface as a result of chemical reactions at the surface from gaseous reactants.

**Chromatogram** -- A plot of detector response against peak volume of solution (eluate) emerging from the system for each of the constituents which have been separated.

**Circuit** -- One complete traverse of the fiber feed mechanism of a winding machine; one complete traverse of a winding band from one arbitrary point along the winding path to another point on a plane through the starting point and perpendicular to the axis.

**Cocuring** -- The act of curing a composite laminate and simultaneously bonding it to some other prepared surface during the same cure cycle (see **Secondary Bonding**).

**Coefficient of Linear Thermal Expansion** -- The change in length per unit length resulting from a one-degree rise in temperature.

**Coefficient of Variation** -- The ratio of the population (or sample) standard deviation to the population (or sample) mean.

**Collimated** -- Rendered parallel.

**Colloidal particle** -- a dispersed particle with a linear dimension of 5 to 100 nm

**Compatible** -- The ability of different resin systems to be processed in contact with each other without degradation of end product properties. (See **Compatible**, Volume 1, Section 8.1.4)

**Components** --

Critical Component - A component whose failure may result in a catastrophic failure or major financial impact of the entire system.

Primary Component: - A component whose failure may significantly degrade system performance, may significantly damage other components in the system or whose failure has a large cost impact

Secondary Component - A component whose failure slightly degrades system performance, will not cause significant damage to other components in the system or has minimal cost impact.

Other Components - A component whose failure does not impact the near-term system performance but may reduce the long-term durability of other components in the system

**Composite Class** -- As used in the handbook, a major subdivision of composite construction in which the class is defined by the fiber system and the matrix class, e.g., organic-matrix filamentary laminate.

**Composite Material** -- Composites are considered to be combinations of materials differing in composition or form on a macroscale. The constituents retain their identities in the composite; that is, they do not dissolve or otherwise merge completely into each other although they act in concert. Normally, the components can be physically identified and exhibit an interface between one another.

**Compound** -- An intimate mixture of polymer or polymers with all the materials necessary for the finished product.

**Condensation Polymerization** -- This is a special type of step-growth polymerization characterized by the formation of water or other simple molecules during the stepwise addition of reactive groups.

**Confidence Coefficient** -- See **Confidence Interval**.

**Confidence Interval** -- A confidence interval is defined by a statement of one of the following forms:

$$(1) P\{a < \theta\} \leq 1 - \alpha$$

$$(2) P\{\theta < b\} \leq 1 - \alpha$$

$$(3) P\{a < \theta < b\} \leq 1 - \alpha$$

where  $1 - \alpha$  is called the confidence coefficient. A statement of type (1) or (2) is called a one-sided confidence interval and a statement of type (3) is called a two-sided confidence interval. In (1)  $a$  is a lower confidence limit and in (2)  $b$  is an upper confidence limit. With probability at least  $1 - \alpha$ , the confidence interval will contain the parameter  $\theta$ .

**Constituent** -- The basic building block of a composite material. Constituents include, but are not limited to, the fiber, matrix, interphase, particulates and any other additives which retain an identity within the composite material. Constituents are typically characterized by their chemical content, volume or weight fraction within the composite, shape and orientation within the composite.

**Continuous Filament** -- A yarn or strand in which the individual filaments are substantially the same length as the strand.

**Continuous Fiber Ceramic Composites (CFCC)** -- a ceramic matrix composite in which the reinforcing phase(s) consist of continuous filaments, fibers, yarn, or knitted or woven fabric.

**Coupling Agent** -- In fabricating composites, any chemical designed to react with both the reinforcement and matrix phases of a composite material to form or promote a stronger bond at the interface.

**Coverage** -- The measure of the fraction of surface area covered by the braid.

**Crazing** -- Apparent fine cracks at or under the surface of an organic matrix.

**Creel** -- A framework arranged to hold tows, rovings, or yarns so that many ends can be withdrawn smoothly and evenly without tangling.

**Creep** -- The time dependent part of strain resulting from an applied stress.

**Creep, Rate Of** -- The slope of the creep-time curve at a given time.

**Crimp** -- The undulations induced into a braided fabric via the braiding process.

**Crimp Angle** -- The maximum acute angle of a single braided yarn's direction measured from the average axis of tow.

**Crimp Exchange** -- The process by which a system of braided yarns reaches equilibrium when put under tension or compression.

**Critical Value(s)** -- When testing a one-sided statistical hypothesis, a critical value is the value such that, if the test statistic is greater than (less than) the critical value, the hypothesis is rejected. When testing a two-sided statistical hypothesis, two critical values are determined. If the test statistic is either less than the smaller critical value or greater than the larger critical value, then the hypothesis is rejected. In both cases, the critical value chosen depends on the desired risk (often 0.05) of rejecting the hypothesis when it is true.

**Crossply** -- Any filamentary laminate which is not uniaxial. Same as Angleply. In some References, the term crossply is used to designate only those laminates in which the laminae are at right angles to one another, while the term angleply is used for all others. In the handbook, the two terms are used synonymously. The reservation of a separate terminology for only one of several basic orientations is unwarranted because a laminate orientation code is used.

**Cumulative Distribution Function** -- See Volume 1, Section 8.1.4.



**Cure, (1)** -- To change the physical properties of a material (usually from liquid to solid) by chemical reaction or by the action of heat or catalysts, alone or in combination, with or without pressure.

**Cure, (2)** -- To irreversibly change, usually at elevated temperatures, the properties of a thermosetting resin by chemical reaction, that is, by condensation, ring closure, or addition. Cure may be accomplished by the addition of curing (cross linking) agents, with or without heat or pressure.

**Cure Cycle**, The time/temperature/pressure cycle used to cure a thermosetting resin system or prepreg.

**Cure Cycle** - The time/temperature/pressure cycle used to cure a thermosetting resin system or prepreg.

**Cure Stress** -- A residual internal stress produced during the curing cycle of composite structures. Normally, these stresses originate when different components of a lay-up have different thermal coefficients of expansion.

**Debond** -- An unbonded or nonadhered region or interface between plies. A separation at the fiber-matrix interface.

**Deformation** -- The change in shape of a specimen caused by the application of a load or force.

**Degradation** -- Changes in the properties and performance, generally brought about by service or aging, that usually reduces the level of performance, utility, safety, reliability, durability, or damage tolerance of the material or component

**Delamination** -- The separation of the layers of material in a laminate. This may be local or may cover a large area of the laminate. It may occur at any time in the cure or subsequent life of the laminate and may arise from a wide variety of causes.

**Denier** -- A direct numbering system for expressing linear density, equal to the mass in grams per 9000 meters of yarn, filament, fiber, or other textile strand.

**Density** -- The mass per unit volume.

**Desorption** -- A process in which an absorbed or adsorbed material is released from another material. Desorption is the reverse of absorption, adsorption, or both.

**Deviation** -- Variation from a specified dimension or requirement, usually defining the upper and lower limits.

**Dielectric Constant** -- The ratio of the capacity of a condenser having a dielectric constant between the plates to that of the same condenser when the dielectric is replaced by a vacuum; a measure of the electrical charge stored per unit volume at unit potential.

**Dielectric Strength** -- The average potential per unit thickness at which failure of the dielectric material occurs.

**Directed Metal Oxidation** -- A method for the formation of ceramic composite matrices by the directed reaction of a molten metal with an oxidant.

**Disbond** -- An area within a bonded interface between two adherends in which an adhesion failure or separation has occurred. It may occur at any time during the life of the structure and may arise from a wide variety of causes. Also, colloquially, an area of separation between two laminae in the finished laminate (in this case the term "delamination" is normally preferred.) (See **Debond, Unbond, Delamination.**)

**Discontinuous Fiber-Reinforced Ceramic Composite** -- a ceramic matrix composite material reinforced by chopped fibers.

**Distribution** -- A formula which gives the probability that a value will fall within prescribed limits. (See **Normal, Weibull, and Lognormal Distributions**, also Volume 1, Section 8.1.4).

**Dry** -- a material condition of moisture equilibrium with a surrounding environment at 5% or lower relative humidity.

**Dry Fiber Area** -- Area of fiber not totally encapsulated by matrix durability.

**Ductility** -- The ability of a material to deform plastically before fracturing.

**Durability** -- A structure's ability to maintain strength and stiffness throughout the service life of the structure.

**Elasticity** -- The property of a material which allows it to recover its original size and shape immediately after removal of the force causing deformation.

**Elongation** -- The increase in gage length or extension of a specimen during a tension test, usually expressed as a percentage of the original gage length.

**Eluate** -- The liquid emerging from a column (in liquid chromatography).

**Eluent** -- The mobile phase used to sweep or elute the sample (solute) components into, through, and out of the column.

**End** -- A single fiber, strand, roving or yarn being or already incorporated into a product. An end may be an individual warp yarn or cord in a woven fabric. In referring to aramid and glass fibers, an end is usually an untwisted bundle of continuous filaments.

**Extensometer** -- A device for measuring linear strain.

**F-Distribution** -- See Volume 1, Section 8.1.4.

**Fabric, Nonwoven** -- A textile structure produced by bonding or interlocking of fibers, or both, accomplished by mechanical, chemical, thermal, or solvent means, and combinations thereof.

**Fabric, Woven** -- A generic material construction consisting of interlaced yarns or fibers, usually a planar structure. Specifically, as used in this handbook, a cloth woven in an established weave pattern from advanced fiber yarns and used as the fibrous constituent in an advanced composite lamina. In a fabric lamina, the warp direction is considered the longitudinal direction, analogous to the filament direction in a filamentary lamina.

**Fell** -- The point of braid formation, which is defined as the point at which the yarns in a braid system cease movement relative to each other.

**Fiber** -- A general term used to refer to filamentary materials. Often, fiber is used synonymously with filament. It is a general term for a filament of finite length. A unit of matter, either natural or manmade, which forms the basic element of fabrics and other textile structures.

**Fiber Content** -- The amount of fiber present in a composite. This is usually expressed as a percentage volume fraction or weight fraction of the composite.

**Fiber Count** -- The number of fibers per unit width of ply present in a specified section of a composite.

**Fiber Direction** -- The orientation or alignment of the longitudinal axis of the fiber with respect to a stated Reference axis.

**Fiber System** -- The type and arrangement of fibrous material which comprises the fiber constituent of an advanced composite. Examples of fiber systems are collimated filaments or filament yarns, woven fabric, randomly oriented short-fiber ribbons, random fiber mats, whiskers, etc.

**Filament** -- a long flexible thread of small cross section, usually extruded or drawn.

**Filamentary Composites** -- A major form of advanced composites in which the fiber constituent consists of continuous filaments. Specifically, a filamentary composite is a laminate comprised of a number of laminae, each of which consists of a nonwoven, parallel, uniaxial, planar array of filaments (or filament yarns) embedded in the selected matrix material. Individual laminae are directionally oriented and combined into specific multiaxial laminates for application to specific envelopes of strength and stiffness requirements.

**Filament Winding** -- A reinforced-plastics process that employs a series of continuous, resin-impregnated fibers applied to a mandrel in a predetermined geometrical relationship under controlled tension.

**Filament Wound** -- Pertaining to an object created by the filament winding method of fabrication.

**Fill (Filling)** -- In a woven fabric, the yarn running from selvage to selvage at right angles to the warp.

**Filler** -- A relatively inert substance added to a material to alter its physical, mechanical, thermal, electrical, and other properties or to lower cost. Sometimes the term is used specifically to mean particulate additives.

**Finish (or Size System)** -- A material, with which filaments are treated, which contains a coupling agent to improve the bond between the filament surface and the resin matrix in a composite material. In addition, finishes often contain ingredients which provide lubricity to the filament surface, preventing abrasive damage during handling, and a binder which promotes strand integrity and facilitates packing of the filaments.

**First Matrix Cracking:** -- The first measurable indication of microcracking of the matrix.

**Fixed Effect** -- A systematic shift in a measured quantity due to a particular level change of a treatment or condition. (See Volume 1, Section 8.1.4.)

**Flash** -- Excess material which forms at the parting line of a mold or die, or which is extruded from a closed mold.

**Former Plate** -- A die attached to a braiding machine which helps to locate the fell.

**Fracture Ductility** -- The true plastic strain at fracture.

**Gage Length** -- the original length of that portion of the specimen over which strain or change of length is determined.

**Gel** -- The initial jelly-like solid phase that develops during formation of a resin from a liquid. Also, a semi-solid system consisting of a network of solid aggregates in which liquid is held.

**Gel Coat** -- A quick-setting resin used in molding processes to provide an improved surface for the composite; it is the first resin applied to the mold after the mold-release agent.

**Gel Point** -- The stage at which a liquid begins to exhibit pseudo-elastic properties. (This can be seen from the inflection point on a viscosity-time plot.)

**Gel Time** -- The period of time from a pre-determined starting point to the onset of gelation (gel point) as defined by a specific test method.

**Glass** -- An inorganic product of fusion which has cooled to a rigid condition without crystallizing. In the handbook, all Reference to glass will be to the fibrous form as used in filaments, woven fabric, yarns, mats, chopped fibers, etc.

**Glass Cloth** -- Conventionally-woven glass fiber material (see **Scrim**).

**Glass Fibers** -- A fiber spun from an inorganic product of fusion which has cooled to a rigid condition without crystallizing.

**Glass Transition** -- The reversible change in an amorphous polymer or in amorphous regions of a partially crystalline polymer from (or to) a viscous or rubbery condition to (or from) a hard and relatively brittle one.

**Glass Transition Temperature** -- The approximate midpoint of the temperature range over which the glass transition takes place.

**Graphite Fibers** -- See **Carbon Fibers**.

**Greige** -- Fabric that has received no finish.

**Hand Lay-up** -- A process in which components are applied either to a mold or a working surface, and the successive plies are built up and worked by hand.

**Hardness** -- Resistance to deformation; usually measured by indentation. Types of standard tests include Brinell, Rockwell, Knoop, and Vickers.

**Heat Cleaned** -- Fibers which have been exposed to elevated temperatures to remove preliminary sizings or binders which are not compatible to processing.

**Heterogeneous** -- Descriptive term for a material consisting of dissimilar constituents separately identifiable; a medium consisting of regions of unlike properties separated by internal boundaries. (Note that all nonhomogeneous materials are not necessarily heterogeneous).

**Homogeneous** -- Descriptive term for a material of uniform composition throughout; a medium which has no internal physical boundaries; a material whose properties are constant at every point, in other words, constant with respect to spatial coordinates (but not necessarily with respect to directional coordinates).

**Horizontal Shear** -- Sometimes used to indicate interlaminar shear. This is not an approved term for use in this handbook.

**Hot pressing** - a fabrication of a ceramic component utilizing temperature and uniaxial pressure to achieve the desired density and shape formation. Densification is achieved by particle rearrangement, viscous/plastic flow, or diffusional transport.

**Humidity, Relative** -- The ratio of the pressure of water vapor present to the pressure of saturated water vapor at the same temperature.

**Hybrid** -- A composite laminate comprised of laminae of two or more composite material systems. Or, a combination of two or more different fibers such as carbon and glass or carbon and aramid into a structure (tapes, fabrics and other forms may be combined).

**Hygroscopic** -- Capable of absorbing and retaining atmospheric moisture.

**Hysteresis** -- The energy absorbed in a complete cycle of loading and unloading.

**Inclusion** -- A physical and mechanical discontinuity occurring within a material or part, usually consisting of solid, encapsulated foreign material. Inclusions are often capable of transmitting some structural stresses and energy fields, but in a noticeably different manner from the parent material.

**Integral Composite Structure** -- Composite structure in which several structural elements, which would conventionally be assembled by bonding or with mechanical fasteners after separate fabrication, are instead laid up and cured as a single, complex, continuous structure; e.g., spars, ribs, and one stiffened cover of a wing box fabricated as a single integral part. The term is sometimes applied more loosely to any composite structure not assembled by mechanical fasteners.

**Interface** -- The boundary between the individual, physically distinguishable constituents of a composite.

**Interlaminar** -- Descriptive term pertaining to some object (e.g., voids), event (e.g., fracture), or potential field (e.g., shear stress) Referenced as existing or occurring between two or more adjacent laminae.

**Interlaminar Shear** -- Shearing force tending to produce a relative displacement between two laminae in a laminate along the plane of their interface.

**Intermediate Bearing Stress** -- The bearing stress at the point on the bearing load-deformation curve where the tangent is equal to the bearing stress divided by a designated percentage (usually 4%) of the original hole diameter.

**Interphase** -- The interface region for a continuous fiber reinforced ceramic matrix composite is the volume between the fiber and the matrix whose degree of bonding is controlled to yield composite-like graceful fiber. Fiber coatings in the interface region are required for most, but not all, ceramic composite systems. For particulate, whisker, and platelet reinforced composites, interface coatings may be necessary to strengthen, toughen, or enhance the thermophysical properties.

**Intralaminar** -- Descriptive term pertaining to some object (e.g., voids), event (e.g., fracture), or potential field (e.g., temperature gradient) existing entirely within a single lamina without Reference to any adjacent laminae.

**Isotropic** -- Having uniform properties in all directions. The measured properties of an isotropic material are independent of the axis of testing.

**Jammed State** -- The state of a braided fabric under tension or compression where the deformation of the fabric is dominated by the deformation properties of the yarn.

**Knitting** -- A method of constructing fabric by interlocking series of loops of one or more yarns.

**Knuckle Area** -- The area of transition between sections of different geometry in a filament wound part.

**k-Sample Data** -- A collection of data consisting of values observed when sampling from k batches.

**Laid-In Yarns** -- A system of longitudinal yarns in a triaxial braid which are inserted between the bias yarns.

**Lamina** -- A single ply or layer in a laminate made up of a series of layers.

**Laminae** -- Plural of lamina.

**Laminate** -- A product made by bonding together two or more layers or laminae of material or materials.

**Laminate Orientation** -- The configuration of a crossplied composite laminate with regard to the angles of crossplying, the number of laminae at each angle, and the exact sequence of the lamina lay-up.

**Lattice Pattern** -- A pattern of filament winding with a fixed arrangement of open voids.

**Lay-up** -- A process of fabrication involving the assembly of successive layers of resin-impregnated material.

**Liquid Infiltration** -- Densification of a composite by infiltration with a liquid and the conversion of the liquid to a solid through controlled processing to develop the desired matrix composition, density, and properties.

**Lognormal Distribution** -- A probability distribution for which the probability that an observation selected at random from this population falls between  $a$  and  $b$  ( $0 < a < b < B$ ) is given by the area under the normal distribution between  $\log a$  and  $\log b$ . The common (base 10) or the natural (base  $e$ ) logarithm may be used. (See Volume 1, Section 8.1.4.)

**Lower Confidence Bound** -- See **Confidence Interval**.

**Macro** -- In relation to composites, denotes the gross properties of a composite as a structural element but does not consider the individual properties or identity of the constituents.

**Macrostrain** -- The mean strain over any finite gage length of measurement which is large in comparison to the material's characteristic distance.

**Mandrel** -- A form fixture or male mold used for the base in the production of a part by lay-up, filament winding or braiding.

**Mat** -- A fibrous material consisting of randomly oriented chopped or swirled filaments loosely held together with a binder.

**Material Acceptance** -- The testing of incoming material to ensure that it meets requirements.

**Material Qualification** -- The procedures used to accept a material by a company or organization for production use.

**Material System** -- A specific composite material made from specifically identified constituents in specific geometric proportions and arrangements and possessing of numerically defined properties.

**Material System Class** -- As used in this handbook, a group consisting of material systems categorized by the same generic constituent materials, but without defining the constituents uniquely.

**Material Variability** -- A source of variability due to the spatial and consistency variations of the material itself and due to variation in its processing. (See Volume 1, Section 8.1.4.)

**Matrix** -- The essentially homogeneous material in which the fiber system of a composite is embedded.

**Matrix Cracking** - Cracking that is completely contained within the matrix material, and do not pass through the reinforcement.

**Matrix Starved Area** -- Area of composite part where the matrix has a non-continuous smooth coverage of the fiber.

**Mean** -- See **Sample Mean** and **Population Mean**.

**Mechanical Properties** -- The properties of a material that are associated with elastic and inelastic reaction when force is applied, or the properties involving the relationship between stress and strain.

**Median** -- See **Sample Median** and **Population Median**.

**Melt Infiltration** -- Densification of a preheated preform/composite by infiltration with a hot liquid which solidifies upon cooling or by in-situ chemical reaction.

**Micro** -- In relation to composites, denotes the properties of the constituents, i.e., matrix and reinforcement and interface only, as well as their effects on the composite properties.

**Microcracks** -- Cracks of limited dimensions, usually of micro-extent.

Note - in heterogeneous systems, microcracks are typically caused by thermal residual stress or by local differences in response to global mechanical, chemical, or thermal applied conditions. Typical microcracks are found as matrix cracks in composites that extend through a ply thickness, parallel to the fiber orientation in that ply, but in ceramic composites, these cracks may also travel perpendicular to the reinforcing fibers and extend through as fiber bundle (or tow), or even through the total thickness of a specimen or component sheet.

**Microstrain** -- The strain over a gage length comparable to the material's characteristic distance.

**Modulus, Chord** -- The slope of the chord drawn between any two specified points on the stress-strain curve.

**Modulus, initial** -- The slope of the initial straight portion of a stress-strain curve.

**Modulus, Secant** -- The slope of the secant drawn from the origin to any specified point on the stress-strain curve.

**Modulus, Tangent** -- The ratio of change in stress to change in strain derived from the tangent to any point on a stress-strain curve.

**Modulus, Young's** -- The ratio of change in stress to change in strain below the proportional limit of a material. (Applicable to tension and compression).

**Modulus of Rigidity** (also Shear Modulus or Torsional Modulus) -- The ratio of stress to strain below the proportional limit for shear or torsional stress.

**Modulus of Rupture, in Bending** -- The maximum tensile or compressive stress (whichever causes failure) value in the extreme fiber of a beam loaded to failure in bending. The value is computed from the flexure equation:

$$F^b = \frac{Mc}{I} \quad 1.7(a)$$

where  $M$  = maximum bending moment computed from the maximum load and the original moment arm,  
 $c$  = initial distance from the neutral axis to the extreme fiber where failure occurs,  
 $I$  = the initial moment of inertia of the cross section about its neutral axis.

**Modulus of Rupture, in Torsion** -- The maximum shear stress in the extreme fiber of a member of circular cross section loaded to failure in torsion calculated from the equation:

$$F^s = \frac{Tr}{J} \quad 1.7(b)$$

where  $T$  = maximum twisting moment,  
 $r$  = original outer radius,  
 $J$  = polar moment of inertia of the original cross section.

**Moisture Content** -- The amount of moisture in a material determined under prescribed condition and expressed as a percentage of the mass of the moist specimen, i.e., the mass of the dry substance plus the moisture present.

**Moisture Equilibrium** -- The condition reached by a sample when it no longer takes up moisture from, or gives up moisture to, the surrounding environment.

**Mold Release Agent** -- A lubricant applied to mold surfaces to facilitate release of the molded article.

**Molded Edge** -- An edge which is not physically altered after molding for use in final form and particularly one which does not have fiber ends along its length.

**Molding** -- The forming of a polymer or composite into a solid mass of prescribed shape and size by the application of pressure and heat.

**Monolayer** -- The basic laminate unit from which crossplied or other laminates are constructed.

**Monomer** -- A compound consisting of molecules each of which can provide one or more constitutional units.

**NDE** -- Nondestructive evaluation. Broadly considered synonymous with NDI.

**NDI** -- Nondestructive inspection. A process or procedure for determining the quality or characteristics of a material, part, or assembly without permanently altering the subject or its properties.

**NDT** -- Nondestructive testing. Broadly considered synonymous with NDI.

**Necking** -- A localized reduction in cross-sectional area which may occur in a material under tensile stress.

**Negatively Skewed** -- A distribution is said to be negatively skewed if the distribution is not symmetric and the longest tail is on the left.

**Nominal Specimen Thickness** -- The nominal ply thickness multiplied by the number of plies.

**Nominal Value** -- A value assigned for the purpose of a convenient designation. A nominal value exists in name only.



**Normal Distribution** -- A two parameter ( $\mu, \sigma$ ) family of probability distributions for which the probability that an observation will fall between a and b is given by the area under the curve

$$f(x) = \frac{1}{\sigma\sqrt{2\pi}} \exp\left[-\frac{(x-\mu)^2}{2\sigma^2}\right] \quad (1.7c)$$

between a and b. (See Volume 1, Section 8.1.4.)

**Normalization** -- A mathematical procedure for adjusting raw test values for fiber-dominated properties to a single (specified) fiber volume content.

**Normalized Stress** -- Stress value adjusted to a specified fiber volume content by multiplying the measured stress value by the ratio of specimen fiber volume to the specified fiber volume. This ratio may be obtained directly by experimentally measuring fiber volume, or indirectly by calculation using specimen thickness and fiber areal weight.

**Observed Significance Level (OSL)** -- The probability of observing a more extreme value of the test statistic when the null hypotheses is true.

**Offset Shear Strength** --- (from valid execution of a material property shear response test) the value of shear stress at the intersection between a line parallel to the shear chord modulus of elasticity and the shear stress/strain curve, where the line has been offset along the shear strain axis from the origin by a specified strain offset value.

**Oligomer** -- A polymer consisting of only a few monomer units such as a dimer, trimer, etc., or their mixtures.

**One-Sided Tolerance Limit Factor** -- See **Tolerance Limit Factor**.

**Orthotropic** -- Having three mutually perpendicular planes of elastic symmetry.

**Oven Dry** -- The condition of a material that has been heated under prescribed conditions of temperature and humidity until there is no further significant change in its mass.

**PAN Fibers** -- Reinforcement fiber derived from the controlled pyrolysis of poly(acrylonitrile) fiber.

**Parallel Laminate** -- A laminate of woven fabric in which the plies are aligned in the same position as originally aligned in the fabric roll.

**Parallel Wound** -- A term used to describe yarn or other material wound into a flanged spool.

**Particulate Reinforced Ceramic Matrix Composites** -- a ceramic matrix composite in which the reinforcing components are particles of equiaxed or platelet geometry (in contrast to whiskers or short fibers)

**Peel Ply** -- A layer of matrix free material used to protect a laminate for later secondary bonding.

**pH** -- A measure of acidity or alkalinity of a solution, with neutrality represented by a value of 7, with increasing acidity corresponding to progressively smaller values, and increasing alkalinity corresponding to progressively higher values.

**Pick Count** -- The number of filling yarns per inch or per centimeter of woven fabric.

**Plied Yarn** -- A yarn formed by twisting together two or more single yarns in one operation.

**Poisson's Ratio** -- The absolute value of the ratio of transverse strain to the corresponding axial strain resulting from uniformly distributed axial stress below the proportional limit of the material.

**Polymer** -- An organic material composed of molecules characterized by the repetition of one or more types of monomeric units.

**Polymer Infiltration/Pyrolysis** -- Densification of a composite by the infiltration of a thermoset polymer, the curing of the polymer, and the subsequent thermal treatment to convert the cured polymer to a ceramic matrix.

**Polymerization** -- A chemical reaction in which the molecules of monomers are linked together to form polymers via two principal reaction mechanisms. Addition polymerizations proceed by chain growth and most condensation polymerizations through step growth.

**Population** -- The set of measurements about which inferences are to be made or the totality of possible measurements which might be obtained in a given testing situation. For example, "all possible ultimate tensile strength measurements for carbon/epoxy system A, conditioned at 95% relative humidity and room temperature". In order to make inferences about a population, it is often necessary to make assumptions about its distributional form. The assumed distributional form may also be referred to as the population. (See Volume 1, Section 8.1.4.)

**Population Mean** -- The average of all potential measurements in a given population weighted by their relative frequencies in the population. (See Volume 1, Section 8.1.4.)

**Population Median** -- That value in the population such that the probability of exceeding it is 0.5 and the probability of being less than it is 0.5. (See Volume 1, Section 8.1.4.)

**Population Variance** -- A measure of dispersion in the population.

**Porosity** -- A condition of trapped pockets of air, gas, or vacuum within a solid material, usually expressed as a percentage of the total nonsolid volume to the total volume (solid plus nonsolid) of a unit quantity of material.

**Positively Skewed** -- A distribution is said to be positively skewed if the distribution is not symmetric and the longest tail is on the right.

**Postcure** -- Additional elevated temperature cure, usually without pressure, to improve final properties.

**Precision** -- The degree of agreement within a set of observations or test results obtained. Precision involves repeatability and reproducibility.

**Preceramic Polymer** -- inorganic or organometallic polymers that can be converted (after polymer curing) to a ceramic by a thermal treatment.

**Precursor** (for Carbon or Graphite Fiber) -- Either the PAN or pitch fibers from which carbon and graphite fibers are derived.

**Precursor (to Ceramic Matrix)** -- A polymeric material which, upon exposure to appropriate processing conditions, is transformed into a ceramic.

**Preform** -- A preshaped mat or woven structure formed from fibers, whiskers or particulate to the desired configuration and reinforcement architecture that is used for subsequent processing.

**Preply** -- Layers of prepreg material, which have been assembled according to a user specified stacking sequence.

**Prepreg** -- Ready to mold or cure material in sheet form which may be tow, tape, cloth, or mat impregnated with resin. It may be stored before use.

**Pressure** -- The force or load per unit area.

**Probability Density Function** -- See Volume 1, Section 8.1.4.

**Proportional Limit** -- The maximum stress that a material is capable of sustaining without any deviation from the proportionality of stress to strain (also known as Hooke's law).

**Quasi-Isotropic Laminate** -- A laminate approximating isotropy by orientation of plies in several or more directions.

**Random Effect** -- A shift in a measured quantity due to a particular level change of an external, usually uncontrollable, factor. (See Volume 1, Section 8.1.4.)

**Random Error** -- That part of the data variation that is due to unknown or uncontrolled factors and that affects each observation independently and unpredictably. (See Volume 1, Section 8.1.4.)

**Reduction of Area** -- The difference between the original cross sectional area of a tension test specimen and the area of its smallest cross section, usually expressed as a percentage of the original area.

**Refractive Index** - The ratio of the velocity of light (of specified wavelength) in air to its velocity in the substance under examination. Also defined as the sine of the angle of incidence divided by the sine of the angle of refraction as light passes from air into the substance.

**Reliability** - Measure of consistency of properties.

**Release Agent** -- See **Mold Release Agent**.

**Resilience** -- A property of a material which is able to do work against restraining forces during return from a deformed condition.

**Resin** -- An organic polymer or prepolymer used as a matrix to contain the fibrous reinforcement in a composite material or as an adhesive. This organic matrix may be a thermoset or a thermoplastic, and may contain a wide variety of components or additives to influence; handleability, processing behavior and ultimate properties.

**Resin Content** -- The amount of matrix present in a composite either by percent weight or percent volume.

**Resin System** -- A mixture of resin, with ingredients such as catalyst, initiator, diluents, etc. required for the intended processing and final product.

**Room Temperature Ambient (RTA)** -- 1) an environmental condition of  $73\pm 5^{\circ}\text{F}$  ( $23\pm 3^{\circ}\text{C}$ ) at ambient laboratory relative humidity; 2) a material condition where, immediately following consolidation/cure, the material is stored at  $73\pm 5^{\circ}\text{F}$  ( $23\pm 3^{\circ}\text{C}$ ) and at a maximum relative humidity of 60%.

**Roving** -- A number of strands, tows, or ends collected into a parallel bundle with little or no twist. In spun yarn production, an intermediate state between sliver and yarn. (Same as tow.)

**S-Basis (or S-Value)** -- The mechanical property value which is usually the specified minimum value of the appropriate government specification or SAE Aerospace Material Specification for this material.

**Sample** -- A small portion of a material or product intended to be representative of the whole. Statistically, a sample is the collection of measurements taken from a specified population. (See Volume 1, Section 8.1.4.)

**Sample Mean** -- The arithmetic average of the measurements in a sample. The sample mean is an estimator of the population mean. (See Volume 1, Section 8.1.4.)

**Sample Median** -- Order the observation from smallest to largest. Then the sample median is the value of the middle observation if the sample size is odd; the average of the two central observations if  $n$  is even. If the population is symmetric about its mean, the sample median is also an estimator of the population mean. (See Volume 1, Section 8.1.4.)

**Sample Standard Deviation** -- The square root of the sample variance. (See Volume 1, Section 8.1.4.)

**Sample Variance** -- The sum of the squared deviations from the sample mean, divided by  $n-1$ . (See Volume 1, Section 8.1.4.)

**Sandwich Construction** -- A structural panel concept consisting in its simplest form of two relatively thin, parallel sheets of structural material bonded to, and separated by, a relatively thick, light-weight core.

**Saturation** -- An equilibrium condition in which the net rate of absorption under prescribed conditions falls essentially to zero.

**Scrim** (also called **Glass Cloth, Carrier**) -- A low cost fabric woven into an open mesh construction, used in the processing of tape or other B-stage material to facilitate handling.

**Secondary Bonding** -- The joining together, by the process of adhesive bonding, of two or more already-cured composite parts, during which the only chemical or thermal reaction occurring is the curing of the adhesive itself.

**Selvage or Selvedge** -- The woven edge portion of a fabric parallel to the warp.

**Set** -- The strain remaining after complete release of the force producing the deformation.

**Shear Fracture** (for crystalline type materials) -- A mode of fracture resulting from translation along slip planes which are preferentially oriented in the direction of the shearing stress.

**Shelf Life** -- The length of time a material, substance, product, or reagent can be stored under specified environmental conditions and continue to meet all applicable specification requirements and/or remain suitable for its intended function.

**Short Beam Shear Strength (SBS)** -- a test result from valid execution of ASTM test method D 2344.

**Significant** -- Statistically, the value of a test statistic is significant if the probability of a value at least as extreme is less than or equal to a predetermined number called the significance level of the test.

**Significant Digit** -- Any digit that is necessary to define a value or quantity.

**Size System** -- See **Finish**.

**Sizing** -- A generic term for compounds which are applied to yarns to bind the fiber together and stiffen the yarn to provide abrasion-resistance during weaving. Starch, gelatin, oil, wax, and man-made polymers such as polyvinyl alcohol, polystyrene, polyacrylic acid, and polyacetates are employed.

**Skewness** -- See **Positively Skewed, Negatively Skewed**.

**Sleeving** -- A common name for tubular braided fabric.

**Slenderness Ratio** -- The unsupported effective length of a uniform column divided by the least radius of gyration of the cross-sectional area.

**Slurry Infiltration** -- Densification of a composite/preform by infiltration with a particulate-liquid suspension, followed by drying. Additional heat treatments may also be done to modify the microstructure, density, phase composition, and/or crystal structure.

**Sol** -- a liquid dispersion of colloidal solid particles, commonly between 5 and 100 nm in size.

**Sol-gel** -- processing, the chemical synthesis of oxide ceramics based on the hydrolysis of metal alkoxides to form sols and gels; as liquids, the sols are suitable for casting and infiltration.

**Solute** -- The dissolved material.

**Specific Gravity** -- The ratio of the weight of any volume of a substance to the weight of an equal volume of another substance taken as standard at a constant or stated temperature. Solids and liquids are usually compared with water at 39°F (4°C).

**Specific Heat** -- The quantity of heat required to raise the temperature of a unit mass of a substance one degree under specified conditions.

**Spindle** -- A slender upright rotation rod on a spinning frame, roving frame, twister or similar machine.

**Standard Deviation** -- See **Sample Standard Deviation**.

**Staple** -- Either naturally occurring fibers or lengths cut from filaments.

**Strain** -- the per unit change, due to force, in the size or shape of a body referred to its original size or shape. Strain is a nondimensional quantity, but it is frequently expressed in inches per inch, meters per meter, or percent.

**Strand** -- Normally an untwisted bundle or assembly of continuous filaments used as a unit, including slivers, tow, ends, yarn, etc. Sometimes a single fiber or filament is called a strand.

**Strength** -- the maximum stress which a material is capable of sustaining.

**Stress** -- The intensity at a point in a body of the forces or components of forces that act on a given plane through the point. Stress is expressed in force per unit area (pounds-force per square inch, megapascals, etc.).

**Stress Relaxation** -- The time dependent decrease in stress in a solid under given constraint conditions.

**Stress-Strain Curve (Diagram)** -- A graphical representation showing the relationship between the change in dimension of the specimen in the direction of the externally applied stress and the magnitude of the applied stress. Values of stress usually are plotted as ordinates (vertically) and strain values as abscissa (horizontally).

**Structural Element** -- a generic element of a more complex structural member (for example, skin, stringer, shear panels, sandwich panels, joints, or splices).

**Structured Data** -- See Volume 1, Section 8.1.4.

**Surfacing Mat** -- A thin mat of fine fibers used primarily to produce a smooth surface on an organic matrix composite.

**Symmetrical Laminate** -- A composite laminate in which the sequence of plies below the laminate midplane is a mirror image of the stacking sequence above the midplane.

**Tack** -- Stickiness of the prepreg.

**Tape** -- Prepreg fabricated in widths up to 12 inches wide for carbon and 3 inches for boron. Cross stitched carbon tapes up to 60 inches wide are available commercially in some cases.

**Tenacity** -- The tensile stress expressed as force per unit linear density of the unstrained specimen i.e., grams-force per denier or grams-force per tex.

**Tex** -- A unit for expressing linear density equal to the mass or weight in grams of 1000 meters of filament, fiber, yarn or other textile strand.

**Thermal Conductivity** -- Ability of a material to conduct heat. The physical constant for quantity of heat that passes through unit cube of a substance in unit time when the difference in temperature of two faces is one degree.

**Thermoplastic** -- A plastic that repeatedly can be softened by heating and hardened by cooling through a temperature range characteristic of the plastic, and when in the softened stage, can be shaped by flow into articles by molding or extrusion.

**Thermoset** -- A plastic that is substantially infusible and insoluble after having been cured by heat or other means.

**Tolerance** -- The total amount by which a quantity is allowed to vary.

**Tolerance Limit** -- A lower (upper) confidence limit on a specified percentile of a distribution. For example, the B-basis value is a 95% lower confidence limit on the tenth percentile of a distribution.

**Tolerance Limit Factor** -- The factor which is multiplied by the estimate of variability in computing the tolerance limit.

**Toughness** -- A measure of a material's ability to absorb work, or the actual work per unit volume or unit mass of material that is required to rupture it. Toughness is proportional to the area under the load-elongation curve from the origin to the breaking point.

**Tow** -- A bundle of continuous filaments. Commonly used in referring to man-made fibers, particularly carbon and graphite fibers, in the composites industry.

**Transversely Isotropic** -- Descriptive term for a material exhibiting a special case of orthotropy in which there exists a plane on which the properties are independent of direction.

**Traveller** -- A small piece of the same product (panel, tube, etc.) as the test specimen, used for example to measure moisture content as a result of conditioning.

**Twist** -- The number of turns about its axis per unit of length in a yarn or other textile strand. It may be expressed as turns per inch (tpi) or turns per centimeter (tpcm).

**Twist, Direction of** -- The direction of twist in yarns and other textile strands is indicated by the capital letters S and Z. Yarn has S twist if, when held in a vertical position, the visible spirals or helices around its central axis are in the direction of slope of the central portion of the letter S, and Z twist is in the other direction.

**Typical Basis** -- A typical property value is a sample mean. Note that the typical value is defined as the simple arithmetic mean which has a statistical connotation of 50% reliability with a 50% confidence.

**Ultimate Strength** -- The maximum stress (tensile, compressive or shear) a material can sustain without fracture; determined by dividing the maximum load in such a test by the original cross sectional area of the specimen. The maximum stress (tensile, compressive or shear) a material can sustain without fracture; determined by dividing the maximum load in such a test by the original cross sectional area of the specimen.

**Unbond** -- An area within a bonded interface between two adherends in which the intended bonding action failed to take place. Also used to denote specific areas deliberately prevented from bonding in order to simulate a defective bond, such as in the generation of quality standards specimens. (See **Disbond, Debond**).

**Unidirectional Laminate** -- A laminate with nonwoven reinforcements and all layers laid up in the same direction.

**Unit Cell** -- A representative volume of material that defines all of the characteristics of the material which, when repeated in three dimensions, can be used to construct the global material or element

**Unstructured Data** -- See Volume 1, Section 8.1.4.

**Upper Confidence Limit** -- See **Confidence Interval**.

**Vacuum Bag Molding** -- A process in which the lay-up is cured under pressure generated by drawing a vacuum in the space between the lay-up and a flexible sheet placed over it and sealed at the edges.

**Variance** -- See **Sample Variance**.

**Viscosity** -- The property of resistance to flow exhibited within the body of a material.

**Void** -- A physical and mechanical discontinuity occurring within a material or part which may be two-dimensional (e.g., disbonds, delaminations) or three-dimensional (e.g., vacuum-, air-, or gas-filled pockets). Porosity is an aggregation of micro-voids. Voids are essentially incapable of transmitting structural stresses or nonradiative energy fields. (See **Inclusion**.)

**Warp** -- The longitudinally oriented yarn in a woven fabric (see **Fill**); a group of yarns in long lengths and approximately parallel.

**Weibull Distribution (Two - Parameter)** -- A probability distribution for which the probability that a randomly selected observation from this population lies between a and b ( $0 < a < b < \infty$ ) is given by Equation 1.7(d) where  $\alpha$  is called the scale parameter and  $\beta$  is called the shape parameter. (See Volume 1, Section 8.1.4.)

$$\exp\left[-\left(\frac{a}{\alpha}\right)^\beta\right] - \exp\left[-\left(\frac{b}{\alpha}\right)^\beta\right] \quad 1.7(d)$$

**Wet Lay-up** -- A method of making a reinforced product by applying a liquid resin system while the reinforcement is put in place.

**Wet Strength** -- The strength of an organic matrix composite when the matrix resin is saturated with absorbed moisture. (See **Saturation**).

**Wet Winding** -- A method of filament winding in which the fiber reinforcement is coated with the resin system as a liquid just prior to wrapping on a mandrel.

**Whisker** -- A short single crystal fiber or filament. Whisker diameters range from 1 to 25 microns, with aspect ratios between 100 and 15,000.

**Work Life** -- The period during which a compound, after mixing with a catalyst, solvent, or other compounding ingredient, remains suitable for its intended use.

**Woven Fabric Composite** -- A major form of advanced composites in which the fiber constituent consists of woven fabric. A woven fabric composite normally is a laminate comprised of a number of laminae, each of which consists of one layer of fabric embedded in the selected matrix material. Individual fabric laminae are directionally oriented and combined into specific multiaxial laminates for application to specific envelopes of strength and stiffness requirements.

**Yarn** -- A generic term for strands or bundles of continuous filaments or fibers, usually twisted and suitable for making textile fabric.

**Yarn, Plied** -- Yarns made by collecting two or more single yarns together. Normally, the yarns are twisted together though sometimes they are collected without twist.

**X-Axis** -- In composite laminates, an axis in the plane of the laminate which is used as the 0 degree Reference for designating the angle of a lamina.

**X-Y Plane** -- In composite laminates, the Reference plane parallel to the plane of the laminate.

**Y-Axis** -- In composite laminates, the axis in the plane of the laminate which is perpendicular to the x-axis.

**Z-Axis** -- In composite laminates, the Reference axis normal to the plane of the laminate.



## 2 INTRODUCTION, HISTORY AND OVERVIEW

### 2.1 HISTORY AND OVERVIEW

Composites have been used throughout history, i.e., straw in bricks, metal rod-reinforced concrete, and lightweight aerospace structures. Fiber reinforced polymer matrix composite materials are being introduced in ever-increasing quantities in military systems and have become a key element in the Department of Defense's effort to lighten the force. However, polymer matrix composites have an inherent temperature limitation based on their hydrocarbon structure. The high temperature alternative to high density metals is ceramics, offering weight savings as well high temperature capability and oxidation resistance.

The sensitivity of monolithic ceramics to small flaws and the resulting brittle fracture has severely limited the use of their unique properties (high temperature strength, low density, chemical stability, and wear resistance) in military and aerospace applications. The development of ceramic composites and associated flaw-tolerant microstructures has been a major goal of structural ceramics over the past two decades.

A composite is defined as a material containing two or more distinct phases combined in such a way so that each remains distinct (Section 1.7). Based on this broad definition of a composite, ceramic matrix composites (CMCs) are conveniently separated into two categories: discontinuous reinforced and continuous fiber reinforced CMCs. Discontinuous reinforced CMCs include particulate, platelet, whisker, fiber and in situ reinforced composites (including nanocomposites). CMCs containing discontinuous second phases are, in general, processed by shaping techniques commonly used for monolithic ceramics; i.e., injection molding, slip casting, and tape casting, followed by sintering to densify the composite. By contrast continuous fiber ceramic composites (CFCCs) have required the development of infiltration (vapor, sol, melt, liquid preceramic polymers) methods that enable the densification of various ceramic matrices in continuous fiber lay-ups and/or net shape woven fiber preforms. Continuous fiber reinforced CMCs are further subdivided into carbon fiber reinforced carbon composites, a.k.a. carbon-carbon composites and other composites.

Both discontinuous reinforced and continuous fiber reinforced composites decrease the susceptibility of ceramic components to catastrophic thermal-structural "brittle failure". The mechanisms by which discontinuous reinforced and continuous reinforced CMCs retard crack initiation and/or propagation are dependent on the nature of material reinforcements (geometry, quantity, and distribution). Understandably, the reinforcements in discontinuous and continuous CMCs give rise to inherently different mechanical properties, different advantages, and different end uses. A list of the general advantages of carbon-carbon, continuous reinforced CMCs, and discontinuous reinforced CMCs are presented in Table 2.1.

A ceramic's resistance to small flaws, crack propagation, and failure can be quantified by measuring a property termed fracture toughness. The fracture toughness coupled with sufficient statistical mechanical property data provide designers the information needed for structural analysis and design safety margins of ceramics.

In general, the fracture toughness values of monolithic ceramics excluding transformation toughened materials do not exceed values of  $4.5 \text{ ksi in}^{1/2}$  ( $5 \text{ MPa}\cdot\text{m}^{1/2}$ ). Discontinuous reinforced CMCs have typical values in the range of 6 to  $10.92 \text{ ksi in}^{1/2}$  (7 to  $12 \text{ MPa}\cdot\text{m}^{1/2}$ ). Continuous fiber CMCs reach values around  $18 \text{ ksi in}^{1/2}$  ( $20 \text{ MPa}\cdot\text{m}^{1/2}$ ) and depending on the fiber architecture can approach values similar to good metallic systems,  $27 \text{ ksi in}^{1/2}$  ( $30 \text{ MPa}\cdot\text{m}^{1/2}$ ). Continuous fiber reinforced CMCs and discontinuous CMCs also exhibit significant differences in yield strengths, linear stress-strain proportional limits, and ultimate strengths. Discontinuous whisker, in situ toughened, and particulate nanocomposites ceramics can reach yield strengths and ultimate strengths approaching 1000 MPa.

**TABLE 2.1** *Characteristics of different classes of CMCs.*

<b>Carbon-Carbon</b>	<b>Continuous CMCs</b>	<b>Discontinuous CMCs</b>
Exceptional High Temp Mech Properties	Excellent High Temp Mech. Properties	Excellent High Temp Mech Properties
High Specific Strength and Stiffness High Fracture Toughness	High Specific Strength and Stiffness High Fracture Toughness	High Specific Strength and Stiffness Higher Useful Strengths.
Dimensional Stability Low Thermal Expansion	Dimensional Stability Low Thermal Expansion	Fracture Toughness Good but Inferior to Continuous CMCs
High Thermal Shock Resistance Graceful Failure Modes	Good Thermal Shock Resistance Graceful Failure Mode	Thermal Shock Resistance Inferior to Continuous CMCs
Tailorable Properties Machinability Poor Oxidation Resistance	Oxidation Resistance Machining More Difficult Processing More Complicated and Expensive	Amenable to Cheaper Conventional Processes Machining Expensive

By contrast, yield strengths and ultimate strengths of continuous fiber reinforced CMCs are around 75 MPa and 350 MPa, respectively. The fibers, which are responsible for the high strain-to-failure and hence, the high fracture toughness of continuous fiber reinforced CMCs, also determine the load carrying (yield strength and ultimate strength) capabilities of the composite. The presence and non-uniform distribution of porosity in the matrices in continuous fiber CMCs is virtually impossible to eliminate. These defects, coupled with property differences between the fibers and ceramic matrices, are responsible for what is known as the "matrix cracking strength". In general, the matrix cracking strength and the yield strength of continuous fiber reinforced CMCs are the same. Once loads that cause matrix cracking are exceeded, the load sharing between fibers and matrix changes and the stress-strain relationship becomes nonlinear.

Another liability for non-oxide based matrices and fibers such as C and SiC is the life-limiting nature of oxidative and corrosive environments. For design safety reasons, most non-oxide CMC components are designed to be used below their yield strength, typically around 50 MPa. For short duration applications, the high strain to failure characteristic of continuous fibers permits continuous fiber reinforced CMCs to be used above their proportional limit. It is this damage tolerance property that provides exceptional resistance to thermal shock environments and graceful failure. Most applications of continuous fiber reinforced CMCs to date have involved structural or heat shield components that encounter light mechanical loads in severe thermal shock environments, typical of turbine and rocket engines.

## 2.2 APPLICATIONS

Carbon-carbon composites are a mature technology. The graphite-like matrix maintains its strength to exceptionally high temperatures of 4000°F (2204°C) in non-oxidizing environments. The historical use of carbon-carbon composites can be traced to ICBM rocket nozzles over three decades ago. Over the past decade and a half, carbon-carbon brakes for military and commercial aircraft have developed into the largest single market for CMCs.

The lightweight and high-temperature stiffness-to-weight properties of carbon-carbon have continued to spur funding of military demonstration projects such as solar array, radar antenna and mirror support structures, heat shields, and radiators for spacecraft. Turbine engine components, reentry vehicle nose tips, and hypersonic leading edge articles have also received demonstration funding.

The next large dual-use market on the horizon for carbon-carbon composites is electronic packaging. The development of very high conductivity carbon fibers coupled with lower cost matrix densification techniques offer promise as printed wiring board or multichip module substrates, thermal planes and other heat dissipation components. Many of these applications are summarized in Table 2.2(a).

**TABLE 2.2(a)** *Applications of carbon-carbon composites.*

Applications Sectors	Carbon-Carbon Composites	
MILITARY	<ul style="list-style-type: none"> <li>• Thermally Conductive Space Structures</li> <li>• Heat Sinks for Electronic Packaging</li> <li>• Solar Array Structures</li> <li>• Graphite Replacement</li> <li>• Leading Edge Material</li> <li>• Antenna Window Materials</li> <li>• High Frequency Radar/Mirror Structures</li> </ul>	<ul style="list-style-type: none"> <li>• Space Radiators</li> <li>• Heat Shields</li> <li>• Turbine Engine Exhaust</li> <li>• Hypersonic Aerodynamic Weapons (HAW)</li> <li>• RV Nose Tips</li> </ul>
Commercial/Dual Use	<ul style="list-style-type: none"> <li>• Off Road Vehicle Brakes</li> <li>• Replacement For Graphite Structures</li> <li>• Diesel Pistons and Exhaust Components</li> <li>• Commercial Aircraft Brakes</li> <li>• Electronic Packaging</li> </ul>	<ul style="list-style-type: none"> <li>• High Temp Furnace Components</li> <li>• Nuclear Reactor Components</li> </ul>

Discontinuous CMCs are experiencing the largest market growth in the commercial sector. Sales of whisker and second phase particulate reinforced ceramic cutting tools are expanding at 45% per year. A wide range of abrasive and corrosive wear applications are emerging for these type of composites. Protective sleeves for molten Al mixer shafts, hot gas filters, fire proof floor tiles and a host of other applications are emerging.

Applications for which non carbon-carbon continuous fiber reinforced CMCs are being considered are listed in Table 2.2(b). Most of these applications are thermal-structural environments in turbine and rocket engines where metallic alloys can not meet the performance and/or durability requirements and where carbon-carbon composites would ablate due to oxidation. The afterburner divergent flaps and seals for fighter aircraft turbine engines and divert propulsion and attitude control nozzles for exo-atmospheric interceptor missile rocket engines are prime examples.

The next generation reusable launch vehicle will likely use CMCs for the thrust cells and the ramp of the Aerospike engine. CMCs are presently being evaluated for leading edges, nose section, inlet cowlings, and the nozzle of future hypersonic vehicles. Future turbine engine applications may include combustors, shrouds, stators, vanes, and other augmentor components for turbine engines. Heat shields to protect metallic structures are also being considered in many aircraft applications. Industrial applications

include porous radiant burner tubes, immersion heater tubes, heat exchangers, tube hangers for oil refining, brakes for racing motorcycles, diesel engine valve guides, and/or corrosion resistant containment shells for chemical pumps.

**TABLE 2.2(b)** *Application of continuous fiber CMCs.*

Applications Sectors	Continuous Fiber CMCs		
MILITARY	<ul style="list-style-type: none"> <li>• F414 and F110 Nozzle Flaps and Seals</li> <li>• F117 Aft Deck Heat Shields</li> <li>• Engine Vanes</li> <li>• Flame Holders</li> </ul>	<ul style="list-style-type: none"> <li>• Orbital Transfer Engine Thrusters</li> <li>• Low Cost Large Rocket Thruster (Million Pound) Heavy Lift Launch</li> <li>• Tactical Missile Combustors, Rotors</li> <li>• Divert and Attitude Control Thrusters</li> </ul>	<ul style="list-style-type: none"> <li>• Turboramjet Variable Area Nozzles</li> <li>• Surveillance OTV Thrusters</li> <li>• Hypersonic Leading Edges, Inlet Cowlings and Nozzles</li> <li>• Linear Aerospike Engine, Thrust Cells and Ramp</li> </ul>
Commercial/Dual Use	<ul style="list-style-type: none"> <li>• Heat Exchangers</li> <li>• Radiant Burner Tubes</li> <li>• Land-Based Gas Turbines</li> <li>• Candle Filters</li> </ul>	<ul style="list-style-type: none"> <li>• MDH Air Preheater Tubes</li> <li>• Immersion Heaters</li> <li>• Seal-less Magnetic Pumps</li> <li>• Motorcycle Brakes</li> </ul>	<ul style="list-style-type: none"> <li>• Diesel Components</li> <li>• Valve Guides</li> <li>• Pistons</li> <li>• Turbocharger Rotors</li> </ul>

The rate at which CMCs will be designed into military systems will be driven to a large extent by the degree to which they are enabling technologies; i.e., absolutely needed to meet the performance requirements of future military systems. Even then, performance requirements will be downgraded and design compromises will be imposed if CMC materials are perceived to be too risky.

MIL-HDBK-17 is intended to educate potential end users on the benefits, applications, design principles, fabrication options, characterization and testing protocols, and standards of CMCs. As the definitive guide for testing and property characterization standards, the Handbook is expected to facilitate advancements by the CMC suppliers. This volume of MIL-HDBK-17 provides an initial set of tools to encourage both end users and suppliers to capitalize on future military windows of opportunity.

### 3 PROCESSING, CHARACTERIZATION AND MANUFACTURING

#### 3.1 CMC SYSTEMS, PROCESSING, PROPERTIES AND APPLICATIONS

##### 3.1.1 CMC processing methods

A number of ceramic composites systems have reached the commercial stage of development in which process and properties are defined and available in commercial quantities and geometries. The different systems are described in terms of their fabrication technology – chemical vapor infiltration, directed metal oxidation, polymer-derived ceramics, oxide systems by sintering and hot-pressing, and carbon-carbon composites by pyrolysis or CVD. Current sources of CMCs are listed in Table 3.1.1.

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**TABLE 3.1.1 CMC Manufacturers**

Albany International Techniweave  
 Ceramic Composites Inc.  
 Composite Factory, Inc.  
 Composite Optics Inc. Ceramics (formerly a Dow Corning business unit)  
 General Electric  
 Goodrich - Aircraft wheels and brakes division  
 Hexcel  
 Honeywell Advanced Composites (formerly DuPont Lanxide Composites)  
 Hitco  
 Hyper-Therm  
 Northrop-Grumman  
 Refractory Composites, Inc.  
 Textron, Inc.  
 Ultramet  
 Synterials

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For ceramic composites, a number of historical and developmental fabrication technologies have not yet advanced to commercial availability. These methods are important to the market in terms of background and historical information and also for the potential of future commercialization, depending on technical and economic success. The specific fabrication systems are: sol-gel processing, reaction processing, and fibrous monoliths.

##### 3.1.1.1 Chemical vapor infiltration CMCs

###### 3.1.1.1.1 CVI fabrication technique

CFCCs (continuous fiber-run ceramic composites) fabricated by chemical vapor infiltration (CVI) typically consist of fibers fashioned into a preform, coated with an interface, and infiltrated with a ceramic matrix via CVI (Reference 3.1.1.1.1(a)). The CVI process consists of chemical vapor deposition (CVD) of a ceramic matrix within a porous, fiber-reinforced preform. During this process the CVI reagents are pumped into a furnace containing a heated preform. These gaseous reagents infiltrate the preform and react at the surface of the fibers building up solid matrix material on them and thereby densifying the preform.

In the interest of optimal densification, CVI can be achieved through five different process types defined below (Reference 3.1.1.1.1(a)):

**Isothermal:** Reagents surround a preform held at a constant temperature and enter via diffusion.

**Thermal Gradient:** Reagents contact the cooler portion of the preform and enter via diffusion to the hot portion of the preform where they react.

**Isothermal-Forced Flow:** Reagents are forced to flow through preform which is held at a constant temperature.

**Thermal Gradient-Forced Flow:** Reagents are forced to flow through preform from the cold to hot portion.

**Pulsed Flow:** Reagents flow into and out of the preform by way of a cyclical evacuation and back filling system.

The Isothermal type is the most widely used and only commercially available process (References 3.1.1.1.1(b) through (i)). While the other four types promise benefits over isothermal processing, they are less mature, are part and shape restrictive, and require specialized equipment and controls (Reference 3.1.1.1.1(a)).

In the isothermal process, reagents enter and gaseous reaction products leave the preform via chemical diffusion (Reference 3.1.1.1.1(j)). In order to get economical densification rates, deposition is often conducted at rapid rates which close off the diffusion paths before infiltration is complete (Reference 3.1.1.1.1(a)). Interruption of the CVI process for periodic surface machining is often necessary to re-open diffusion paths (Reference 3.1.1.1.1(a)).

Since CVI is a form of the well-established CVD technique, almost any ceramic compound can be deposited as a CFCC matrix. The most common commercially available CVI matrices are SiC and carbon. Table 3.1.1.1.1 describes these and other possible matrices, the typical fibrous reinforcement used, and their basic chemical reactions.

Some machining may take place between CVI densifications for dimensional control and re-opening of diffusion paths to the interior of the part. The final CVI densification seals all machined edges and completes the part. In some cases, an external environmental coating is applied at this point (Reference 3.1.1.1.1(k)).

The key process steps for a CVI processed CFCC are shown in Figure 3.1.1.1.1. The order and nature of these steps will vary with manufacturer. Preforming techniques are described in Section 3.4. Consolidated preforms are placed inside graphite tooling to retain part shape during processing. Interface coatings are applied to the tooled preform usually through CVI techniques (see Section 3.3). The coated preform with tooling is loaded into a CVI furnace for matrix infiltration. The initial cycle is designed to deposit sufficient matrix to harden the preform and allow removal of the tooling for further CVI densification.

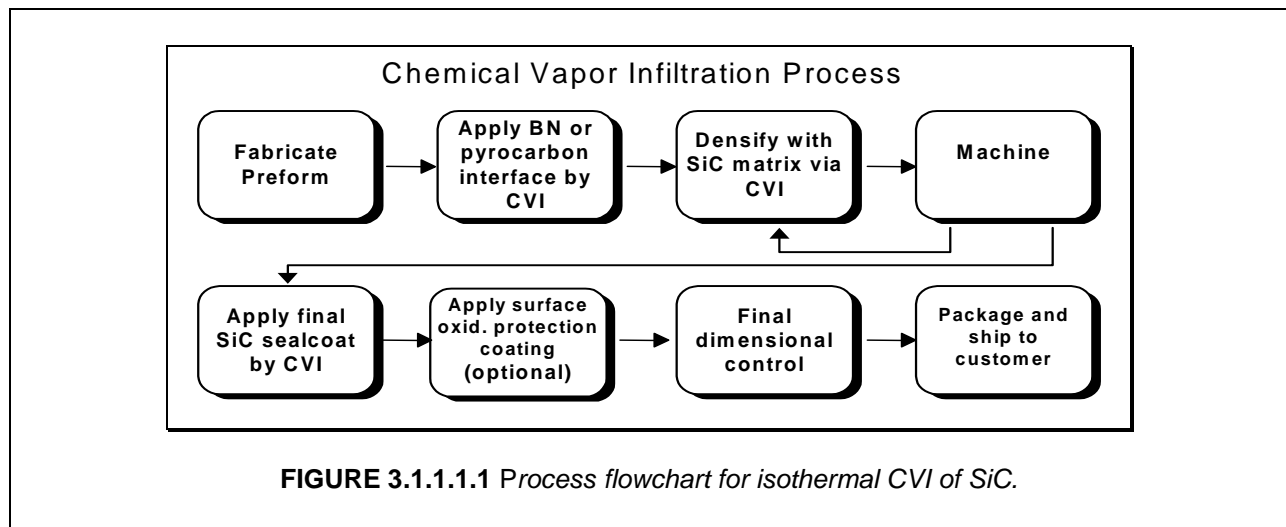
#### 3.1.1.1.2 *Typical properties for CVI CFCCs*

During loading, continuous fiber ceramic composites exhibit linear elastic behavior followed by nonlinear stress-strain response beyond matrix cracking (Reference 3.1.1.1(a)). Current CMCs fabricated by CVI exhibit tensile strength and elongation values of at least 36.3 ksi (250 MPa) and 0.5%, respectively, and can operate up to 2012°F (1100°C). CVI SiC matrix composite turbine blades demonstrated excellent resistance to thermal shock and thermal gradients, and to thermomechanical fatigue (Reference 3.1.1.1.1(a) and 3.1.1.1.2(a)).

CVI processed matrices are superior to other fabrication methods in their purity and microstructure (Reference 3.1.1.1.1(b)). As a result, CVI CFCCs offer enhanced chemical compatibility and corrosion resistance (References 3.1.1.1.2(b) and 3.1.1.1.1(a)).

TABLE 3.1.1.1.1 CVI CFCCs.

Matrix	Fiber/ Reinforcement	Typical Used Reactions
Carbides		
C	carbon	$\text{CH}_4 \rightarrow \text{C} + 2\text{H}_2$
SiC	Nicalon™, Nextel™, carbon, $\text{Al}_2\text{O}_3$ , SiC	$\text{CH}_3\text{SiCl} + \text{H}_2 \rightarrow \text{SiC} + 3\text{HCl}$
TiC	carbon	$\text{TiCl}_4 + \text{CH}_4 + \text{H}_2 \rightarrow \text{TiC} + 4\text{HCl}$
B <sub>4</sub> C	carbon	$4\text{BCl}_3 + \text{CH}_4 + \text{H}_2 \rightarrow \text{B}_4\text{C} + 12\text{HCl}$
ZrC (HfC)	carbon	$\text{ZrCl}_4 + \text{CH}_4 + \text{H}_2 \rightarrow \text{ZrC} + 4\text{HCl}$
Cr <sub>3</sub> C <sub>2</sub>	$\text{Al}_2\text{O}_3$	$\text{CrCl}_x + \text{CH}_4 + \text{H}_2 \rightarrow \text{Cr}_3\text{C}_2 + \text{HCl}$
TaC	carbon	$\text{TaCl}_5 + \text{CH}_4 + \text{H}_2 \rightarrow \text{TaC} + \text{HCl}$
Nitrides		
Si <sub>3</sub> N <sub>4</sub>	Nicalon™, Nextel™, carbon	$3\text{SiCl}_4 + 4\text{NH}_3 + \text{H}_2 \rightarrow \text{Si}_3\text{N}_4 + 12\text{HCl}$
BN	BN, SiO <sub>2</sub> , Nextel™, carbon	$\text{BX}_3 + \text{NH}_3 + \text{H}_2 \rightarrow \text{BN} + 3\text{HX}$ (X=Cl, F)
Borides		
TiB <sub>2</sub>	carbon, Nicalon™, $\text{Al}_2\text{O}_3$	$\text{TiCl}_4 + 2\text{BCl}_3 + \text{H}_2 \rightarrow \text{TiB}_2 + 10\text{HCl}$
Oxides		
ZrO <sub>2</sub>	$\text{Al}_2\text{O}_3$ , mullite, carbon	$\text{ZrCl}_4 + 2\text{CO}_2 + 2\text{H}_2 \rightarrow \text{ZrCl}_4 + 2\text{H}_2\text{O} + 2\text{CO}$ $2\text{CO} \rightarrow \text{ZrO}_2 + 2\text{CO} + 4\text{HCl}$
$\text{Al}_2\text{O}_3$	Nextel™, $\text{Al}_2\text{O}_3$ , carbon	$2\text{AlCl}_4 + 3\text{CO}_2 + 3\text{H}_2 \rightarrow 2\text{AlCl}_4 + 3\text{H}_2\text{O} + 3\text{CO}$ $3\text{CO} \rightarrow \text{Al}_2\text{O}_3 + 3\text{CO} + 6\text{HCl}$



### 3.1.1.1.3 Typical applications for CVI CFCCs

CVI CFCCs are currently being evaluated for many industrial and aerospace applications. Such as:

Product Areas	Examples
Turbine engines (aerospace and stationary)	Combustors, liners, turbine rotors
Heat recovery equipment	Preheaters, recuperators, heat exchangers
Burners	Radiant tube and screen burners
Process equipment	Reformers, reactors, heat exchangers
Waste incineration	Furnace internals, particle separators
Separation/Filtration	Filters, substrates, centrifuges
Structural components	Beams, panels, headers
Aerospace	Thermal protection, thruster nozzles, turbo pump components, leading edges

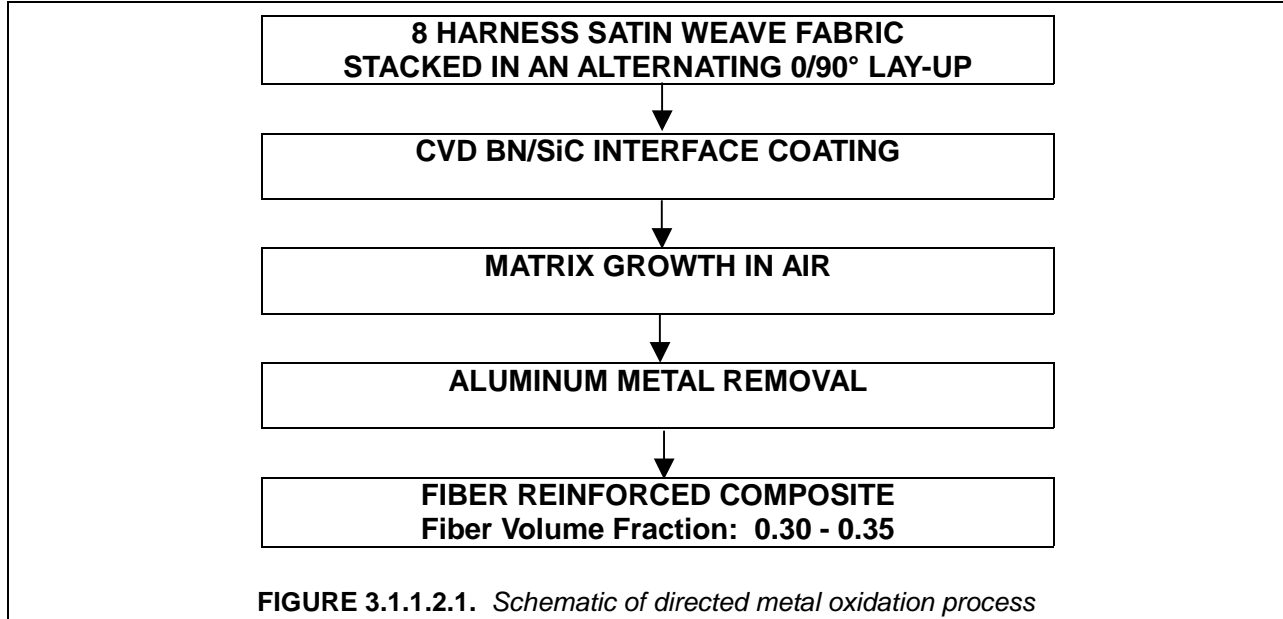
### 3.1.1.2 Directed metal oxidation (DIMOX™)

#### 3.1.1.2.1 Basic processing procedures for DIMOX™

Dimox™ is a method for the formation of ceramic composite matrices by the directed reaction of a molten metal with an oxidant. The most common Dimox™ system is aluminum oxide, however, there are development efforts with the aluminum nitride and silicon nitride systems. The Dimox™ process is proprietary to Honeywell Advanced Composites, Inc., of Newark, Delaware. The basic technique is to directly react a molten metal (aluminum) with an oxidant (air) to form the matrix of a composite. During processing the reaction is sustained by the wicking of liquid metal along interconnected microscopic channels in the aluminum oxide to the free surface promoting outward growth from the original metal surface. The resulting matrix material is typically a three dimensionally interconnected ceramic reaction product with some interconnected metal. Transmission electron microscopy (TEM) studies on aluminum oxide matrices have shown clean grain boundaries, free from low melting point phases that can lead to inferior properties, particularly creep, at elevated temperatures.

Ceramic composites can be formed by placing filler or reinforcement materials (e.g., SiC or Al<sub>2</sub>O<sub>3</sub> fibers, particles, or platelets) in the path of the outward growing reaction product. This forming process is achieved by first shaping the desired reinforcement materials into a preform of the required dimensions. Preforming techniques may involve conventional ceramic processes such as pressing, slip casting, injection molding, etc., in the case of particulates or platelets. Alternatively, fabric lay-up, weaving, braiding, or filament winding might be used for a fiber reinforcement. A growth barrier material is applied to one or more surfaces of the preform to locally stop the matrix growth process and enable fabrication to net or near net shape. The preform and the parent metal alloy are heated to the growth temperature where rapid oxidation of the parent metal alloy occurs outward and into the adjacent preform. For aluminum oxide matrix composites, the preform and aluminum alloy are typically heated to 1652 - 2192°F (900 - 1200°C) in air. Under these conditions, the aluminum oxidizes and a three dimensionally interconnected matrix of alpha aluminum oxide grows into the adjacently placed preform. An interconnected network of microscopic metal channels, which are at most a few microns in diameter, is present in the matrix phase. Driven by surface energy forces, the molten alloy wicks from the aluminum alloy reservoir through the micro-channels to the growth front, where it reacts to continue the matrix growth process. The reaction is terminated when it comes in contact with the gas permeable barrier layer applied to the preform surface, thus enabling fabrication of the composite to the desired shape. Typical growth times for these composites may involve from one to three days of furnace time. A schematic of the directed metal oxidation process is shown in Figure 3.1.1.2.1.





3.1.1.2.2 Typical properties of DIMOX™

Mechanical Properties	Units	Temp 73°F/ 23°C	Temp 2012°F/1100°C
Tensile Strength	ksi / MPa	35.6 / 246	27.5 / 190
Elongation	%	0.62	0.53
Modulus	Msi / GPa	21.0 / 144	18.7 / 129
Proportional Limit	ksi / MPa	9.4 / 65	9.6 / 66

3.1.1.2.3 Typical applications for DIMOX™

Dimox™ CFCCs are currently being evaluated for many industrial and aerospace applications, such as:

Product Areas	Examples
Turbine engines (aerospace and stationary)	Combustors, liners, turbine rotors
Heat recovery equipment	Preheaters, recuperators
Burners	Radiant tube and screen burners
Process equipment	Reformers, reactors, heat exchangers
Separation/Filtration	Filters, substrates, centrifuges
Structural components	Beams, panels, headers

3.1.1.3 Polymer derived ceramics

3.1.1.3.1 Introduction

Polymer derived ceramics offer a unique solution to fabricating ceramic matrix composites. By tailoring polymer molecular structure and molecular weight, materials can be produced that allow fabrication

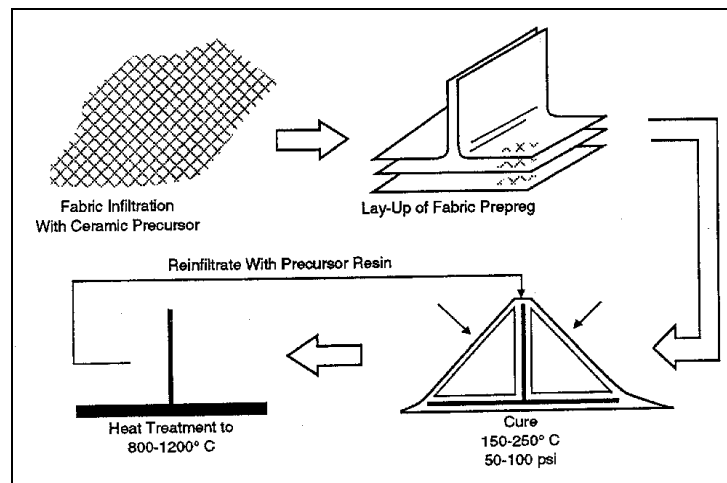
using polymer composite processes such as lay-up-autoclave consolidation, resin transfer molding, filament winding and fiber placement. Using polymers with high char yields, the resulting parts can be heat treated (pyrolyzed) in inert or reactive atmospheres to yield fiber reinforced ceramic matrix composites. This approach has been used to fabricate a variety of ceramic matrix composite compositions, including the commonly available matrices of Si-C, Si-C-O, Si-N, and Si-N-C.

As is the case with other ceramic matrix composites being discussed, coated continuous fibers are introduced into the polymer derived ceramic matrix to provide the desired strength, toughness, and "graceful failure". The coating is designed to properly tailor the fiber-matrix interface to achieve the desired material properties and is similar, both in composition and method of application, to those used with other ceramic matrix composites. Similar fibers are also used with the added ability to incorporate lower temperature capable fibers (Astroquartz, Nextel 312) into a polymer derived ceramic matrix when fabricated at low processing temperatures (<1832°F (<1000°C)).

Particulate ceramics and ceramic whiskers and platelets can be used to supplement the matrix provided by the pyrolyzed polymer. Fillers can improve matrix properties by reducing and disrupting the regularity of matrix cracks that form during the shrinkage of the polymer precursor. In addition, the fillers can enhance the ceramic yield during initial pyrolysis by their mass or by reacting with pyrolysis by-products or the pyrolysis atmosphere and can be used to strengthen and toughen the matrix. Well-chosen fillers can slightly raise composite modulus and improve interlamina properties.

### 3.1.1.3.2 Fabrication

The general process for producing ceramic matrix composites using polymer pyrolysis is illustrated in Figure 3.1.1.3.2(a). The process consists of: (a) low temperature shape making, (b) high temperature pyrolysis, and (c) repeated impregnation-pyrolysis to achieve suitable density. The preceramic polymer process to ceramic matrix composite thus is very similar to the process utilized for polymer composites. In principle any process that has been used for polymer composite shape making can be utilized. The densification cycles are nominally similar to those used for carbon-carbon although they are operationally much simpler because of less toxic, easier to use polymers, and shorter pyrolysis cycles. Pyrolysis conditions vary somewhat but all systems require inert atmosphere pyrolysis to at least 1292°F (700°C) (typically 1652-2192°F (900-1200°C)). The number of reimpregnations can vary depending on polymer system and end-use goals, but at least two are needed to get decent mechanical properties. Typically 4 - 10 reimpregnations are required.

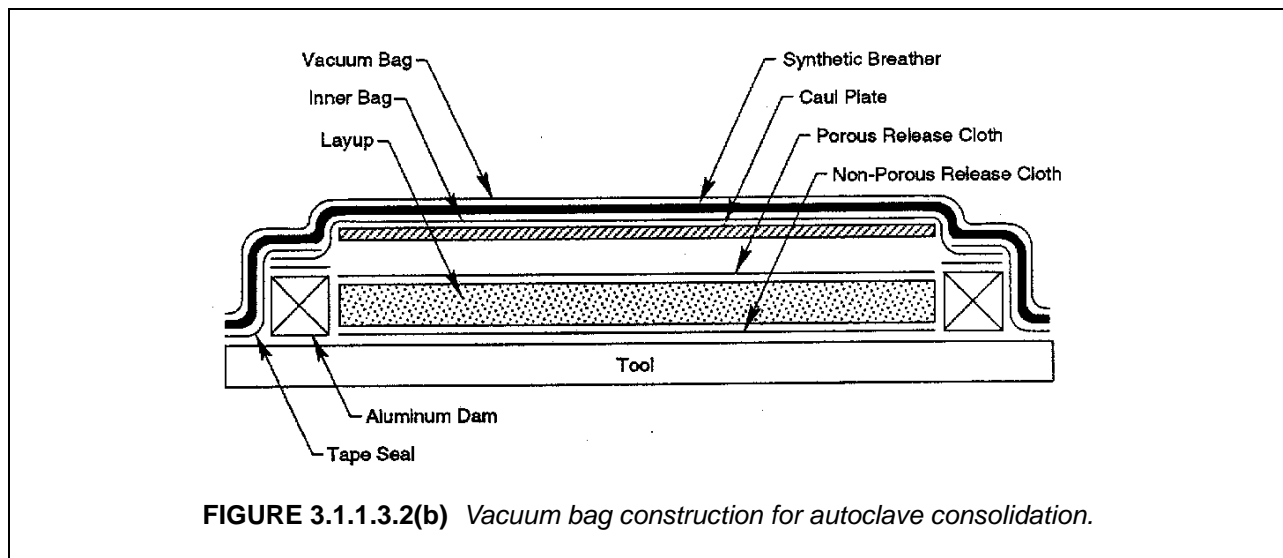


**FIGURE 3.1.1.3.2(a)** Processing of polymer derived ceramics.

A widely used approach to fabricating ceramic matrix composites by polymer pyrolysis uses flat lay-ups of cloth or tape prepreg material, followed by consolidation in an autoclave or a low temperature press. Complex parts are fabricated by wrapping cut pieces of prepreg around appropriate tooling, followed by autoclave consolidation. Typically, a cloth "prepreg" is fabricated by applying preceramic polymer to a woven ceramic cloth. For flat laminate material studies, a harness satin weave is often used with a warp aligned or 0/90 lay-up, obtained by alternate flipping of cloth to get good "nesting" of the weave. The resin used is typically a low molecular weight, solvent soluble, waxy polymer. Polymer is usually applied to the cloth from a concentrated solvent solution. Fine, particulate fillers can be dispersed in this prepreg solution. To achieve good tack, typical number average molecular weights are in the range of 1000-3000.

Often a prepreg of unidirectional fibers is preferred. Such a prepreg consisting of unidirectional tow sheet can be made by running tow through a polymer solution or a polymer-filler slurry. Proper spreading of tow facilitates filler incorporation. The wet tow is then dried and carefully wound on a drum to keep the tows parallel. Prepreg sheets are then cut off the drum. Consolidation of prepreg is best done by autoclave, which can apply uniform pressure to the whole part (a distinct asset for complex shaped parts). Similar time-temperature-pressure conditions can be used to consolidate flat laminates in a simple, electrically heated polymer press (e.g., a Carver™ press). Prepreg patterns for the lay-up are cut from prepregged cloth by covering both sides with Teflon™ coated release paper, cooling the cloth prepreg overnight in a freezer, and cutting patterns in the cold prepreg with templates and a razor blade knife or a roller cutter. The release paper can be peeled off the cold prepreg prior to lay-up. Good prepreg tack is essential when wrapping cloth around complex tooling. If repositioning of cloth or tape is necessary, debonding can be facilitated by cooling the area with a cooling aerosol spray.

A typical autoclave lay-up setup is shown in Figure 3.1.1.3.2(b). For complex shapes, tooling appropriate for fiber-epoxy composites is sufficient. Typically, machined aluminum tools are used. A non-silicone mold release is coated over the tool prior to use. Autoclave cycles vary, based on the type of preceramic polymer being used but basic principles are similar: the polymer must be consolidated above its  $T_g$ , ideally at low melt viscosity ( $\leq 10^3$  poise), but before the polymer begins to thermoset.



Pyrolysis of the initial shape converts polymer to ceramic while generating volatile by-products. Since a large fraction of these gases are low in molecular weight (e.g.,  $H_2$ ,  $CO$ ), even a high ceramic yield prepolymer evolves considerable gas volumes during pyrolysis. Pyrolysis cycles need to be slow, especially

for thicker parts, to allow volatiles to slowly diffuse out of the matrix without causing delamination. Ramp rates need to be tailored so they are slowed in regions where greatest quantities of volatiles are released. This is typically done by consideration of thermal gravimetric data. However, it could best be done by use of mass spectrometric analysis, to better take into account the molecular weights of evolved species. Typical pyrolysis cycles ramp up to temperatures of 1472-2552°F (800-1400°C) over periods of 1-2 days to avoid delamination concerns. As we have discussed previously, one would want to pyrolyze below temperatures where the reinforcing fiber of choice loses strength. Pyrolysis atmosphere is most commonly argon or nitrogen. However, it is known that ammonia atmospheres can result in a pure amorphous silicon nitride with low free carbon content. Such a reactive atmosphere could also result in increased ceramic yields through forming nitrides with reactive fillers. While many parts are pyrolyzed free standing, graphite tooling can be used for parts where concerns with thermal induced distortions exist.

Successful polymer fabrication yields a 100% dense fiber reinforced polymer composite. However, weight loss during pyrolysis, coupled with shrinkage in converting the preceramic polymer with a density of approximately  $1.0\text{g/cm}^3$  to an amorphous ceramic with a density of approximately  $2.0\text{g/cm}^3$ , results in a void content, after initial pyrolysis, of 20-30%. To reduce porosity, reimpregnation of the initial ceramic matrix composite shape is necessary. Reimpregnation is best conducted with a very low viscosity prepolymer. Less successful alternatives use heated polymers to reduce viscosity or reimpregnation with solvent dissolved polymer. Higher viscosity polymers may require pressure impregnation. The reimpregnated low viscosity polymer should thermoset, without volatile generation, prior to pyrolysis and should have a high ceramic yield. Figure 3.1.1.3.2(c) shows typical density versus impregnation number data, as well as illustrating the effect of additional process variables. For example, an early experiment utilizing a poor thermosetting polysilazane is shown. Note the low initial density due to interlaminar expansion during pyrolysis. Two additional experiments are shown with 15% by volume filler in the prepreg. Using this approach, more rapid densification is possible. The unfilled laminate shows typical density versus impregnation number data. In this ceramic matrix composite, approximately 40% by volume in fiber, the initial ceramic density can be calculated as roughly  $1.4\text{g/cm}^3$  assuming fiber and matrix densities of  $2.3$  and  $2.0\text{g/cm}^3$ . Likewise, densities after further densification - pyrolysis cycles can be calculated by assuming complete volume filling of porosity with polymer, a 20% loss of polymer during pyrolysis, and a 50% shrinkage of the remaining matrix during conversion to ceramic.

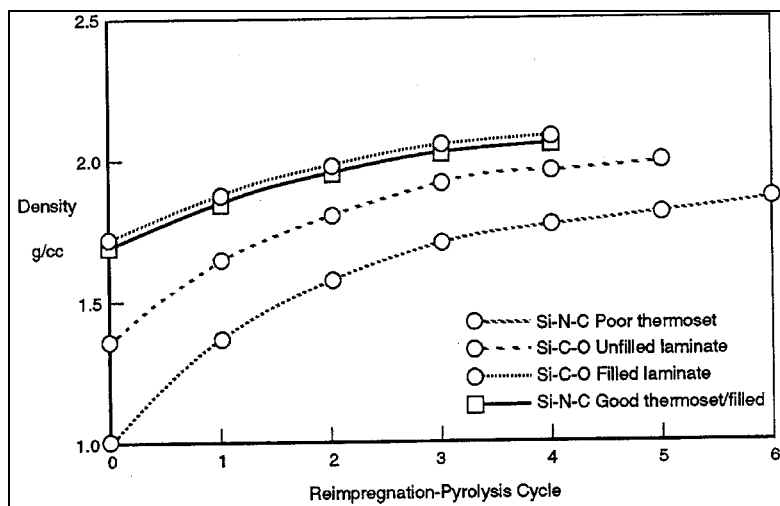


FIGURE 3.1.1.3.2(c) Typical changes in density versus reimpregnation-pyrolysis cycle.

To achieve acceptable tensile properties, at least three impregnations appear to be needed. To improve interlaminar properties, to reduce porosity, and to improve the oxidative stability of an unstable fiber matrix interface, additional reimpregnations are often used. Reimpregnation is best done by immersing the evacuated part in the liquid polymer in a vacuum bag. Alternatively, the part can be immersed in liquid polymer and then put in a bell jar or vacuum furnace until air bubbling stops. If complete immersion is impossible, the polymer can be repeatedly brushed onto the surface of the part. Higher viscosity polymers may require pressure impregnation. Following reimpregnation, the part is warmed in a low temperature oven to cure the reimpregnated polymer, with care taken to reduce evaporation, e.g., cover or wrap the part during cure. The part can then be pyrolyzed as per previous discussions.

Machining of polymer derived ceramic matrix composites can be performed when partially dense or after densification is complete. Typically, a part is machined after 2-3 reimpregnation cycles to ensure adequate strength for handling. At this stage of fabrication, conventional machine tools are frequently used. Due to the low distortion typically present when processing polymer derived ceramic matrix composites, machining is frequently limited to holes, mating surfaces, and trimming. Throughout the manufacturing process, a number of conventional NDE methods, e.g., thermal imaging, X-ray, and ultrasonic, can be used to ensure quality.

#### 3.1.1.4 Carbon-carbon composites

Carbon is an exceptionally stable material in the absence of oxygen, surviving temperatures greater than 3992°F (2200°C) in vacuum and inert conditions. In addition, carbon is a light-weight material with a density of approximately 2.0 g/cm<sup>3</sup>. However monolithic graphite is a brittle, low-strength material that cannot be easily formed into large complex shapes.

To overcome the strength and fabrication limitations, defense and aerospace-funded researchers developed carbon-carbon composites in which high-strength carbon fibers are incorporated into a carbon matrix. The history, development, and general properties of carbon-carbon composites are well-described by Buckley (Reference 3.1.1.4(a)). For high temperature applications carbon-carbon composites offer exceptional thermal stability (>3992°F (>2200°C)) in non-oxidizing atmospheres along with low densities (1.5 - 2.0 g/cm<sup>3</sup>).

From a structural and thermal perspective, the use of high-strength and high-modulus carbon fibers is the key factor in developing mechanical strength and durability while retaining the high temperature capability. Through the use of such fibers, carbon-carbon composites are now used in rocket nozzles, nosecones for reentry vehicles, leading edges, cowlings, heat shields, aircraft brakes, brakes for racing vehicles, and high temperature furnace setters and insulation. These applications utilize the following nominal properties of carbon-carbon composites (which depend on fiber type, fiber architecture, and matrix density).

- Ultimate Tensile Strength > 40,000 psi (266 MPa)
- Modulus of Elasticity > 10 Msi (69 GPa)
- Thermal Conductivity .9-19 Btu-in/(s-ft<sup>2</sup>-°F (6 - >100 W/m·K)
- Thermal Expansion 1.1 ppm/K
- Density < 2 g/cm<sup>3</sup>

These properties are key performance factors in using carbon-carbon composites in structural, low thermal expansion, and thermal management applications. In particular, the low thermal expansion and range of thermal conductivities give carbon-carbon composites high thermal shock resistance.

The one major shortcoming of carbon-carbon composites is their oxidation susceptibility. At temperatures above 932°F (500°C), both the matrix and the fiber are vulnerable to oxidation, if they are not protected from oxygen exposure. The two primary oxidation protection methods are external coatings and internal oxidation inhibitors. Surface coatings provide an external barrier to oxygen penetration. Silicon carbide is a common component of the external coating system. The addition of internal oxidation inhibitors act either as internal barriers to oxygen ingress or as oxygen sinks (forming a protective barrier). The

time-temperature-cycle capabilities of these oxidation barriers are the primary limit to the temperature capabilities of current carbon-carbon composites in oxidation environments. Current temperature limits are on the order of 2912°F (1600°C) for short term exposures.

Carbon-carbon composites are fabricated by infiltrating a carbon fiber preform (braid, weave, fabric, mat) with the carbon matrix. The matrix is developed by liquid-infiltration, chemical vapor infiltration, or a combination of the two methods. Liquid infiltration is done with either a carbon thermosetting precursor resin (such as phenolic or furan resins). The resin is cured and pyrolyzed in an inert atmosphere to convert the resin to a carbon. This infiltration/cure/pyrolysis cycle is repeated to incrementally build up the density, because porosity and cracks form in the matrix component during the pyrolysis cycle. Reinfiltration is also done with thermoplastic carbon sources, such as coal-tar and petroleum pitches. Repeated densification cycles reduce the porosity until the desired open porosity is achieved.

The alternate densification method for carbon-carbon composites is the use of chemical vapor infiltration (Reference 3.1.1.4(b)), in which a hydrocarbon gas (e.g., methane, ethane, propane) is used as the carbon source. The carbon fiber preform is heated to temperatures of approximately 1832-2192°F (1000-1200°C) and the reactant gas is flowed through the preform. The gas pyrolyzes to form carbon in the body of the preform.

The two different methods of densification produce different carbon microstructures. The use of one method or a combination of methods will produce differences in matrix properties, which have an effect on the composite performance. As formed, the carbon (by either resin or CVI) tends to be amorphous or microcrystalline in structure. It is common practice in producing high performance carbon-carbon composites to convert the matrix to highly crystalline graphite by heat treatment in the 3632-5432°F (2000-3000°C) range. This graphitic conversion of the matrix carbon increases the modulus, load-carrying capability, and the thermal conductivity of the composite.

The properties, microstructure, and fabrication of carbon fibers are described in the carbon fiber chapter of this Volume. The mechanical properties of different carbon fibers vary across a broad range, depending on the carbon content, the microstructure and the degree of graphitization. Ultimate tensile strengths range from 200-820 ksi (1.38 - 8.5 GPa) with tensile moduli ranging from 23-120 Msi (150-830 GPa). In a similar manner, the thermal conductivity along the length of the fiber can range from 4-212 Btu-in/(s-ft-°F) (22-1100 W/m·K). The mechanical and thermal properties of a given carbon-carbon composite will depend strongly on the strength and architecture of the reinforcing carbon fibers as well as the composition, density, and crystal structure of the carbon matrix.

#### 3.1.1.5 Oxide systems –sinter/hot press

Reserved for future use.

#### 3.1.1.6 Sol-gel processing

##### 3.1.1.6.1 Introduction

The term sol-gel, a contraction of “solution-gelation,” encompasses a wide variety of chemically synthesized precursors and an array of liquid processing techniques to form ceramics and glasses. Typically a chemical precursor is hydrolyzed, polymerized into a gel, then dried and fired to produce a particular glass or ceramic composition. The precursors range from mixtures of water, alcohols and metal alkoxides to commercially available, stabilized colloids containing discrete ceramic particles as well as solutions containing inorganic polymers derived from metal salts. Johnson in 1985 (Reference 3.1.1.6.1(a)) proposed categorizing the traditional sol-gel processing into (A) polymerized alkoxides and (B) colloidal sols, each of which was then subcategorized. A third category would likely now include inorganic polymeric solutions. Polymerized alkoxides were divided by constituents into (1) all-alkoxide components and (2) alkoxide precursors with some metal salt components added. Colloidal sols were divided into (1) those involving precipitation and peptization and (2) those consisting of a dispersion of colloidal particles. Johnson acknowledged that certain organometallics such as aluminum isopropoxide can be included under

both the “polymerized alkoxide” category and the “colloidal sol” category since the hydrolyzed aluminum isopropoxide can form colloidal particles under certain processing conditions. The broad categories are useful, however, and hold for most examples.

Alkoxide-based processing involves the use of organometallics, such as aluminum sec-butoxide or tetraethyl orthosilicate, which are hydrolyzed and then polymerized into a gel through the action of an acid or base catalyst. Mixtures of alkoxide precursors such as these often require special steps to maintain chemical homogeneity because of differing hydrolysis rates. Techniques for alkoxide-based processing often include laboratory scale preparation utilizing specialty glassware and reflux condensers to produce the sols. There is a variety of literature (Reference 3.1.1.6.1(b) through (e)) relating to formulating alkoxide-based sols, particularly silica and silicate-based glasses. (Often metal salts, typically nitrates, are added to introduce minor constituents. An example of alkoxide-based processing involving the use of nitrates is as follows. Aluminum sec-butoxide and tetraethylorthosilicate (TEOS),  $\text{Si}(\text{C}_2\text{H}_5\text{O})_4$ , can be hydrolyzed and combined with magnesium nitrate to form a magnesium aluminosilicate sol.)

Colloidal solutions for certain oxide systems, such as silica and alumina, are commercially available. Ludox™, manufactured by E.I. DuPont de Nemours & Co. (DuPont), is perhaps the most widely used and consists of spheres of amorphous silica dispersed in an aqueous medium. It is commonly used for fiber reinforced radome manufacture and has gained popularity as an easy to use, high yield silica source when manufacturing sol-gel mullite. Nalco Chemical Company also produces a range of colloidal products including colloidal silica, alumina and alumina-coated silica. Nyacol Products produces alumina, yttria, ceria and zirconia sols.

Inorganic polymeric solutions are currently being utilized by many groups for matrix formation in CFCC's as well as for coating fibers. McDermott Technology, Inc. (MTI) makes an inorganic alumina polymeric from aluminum nitrate. MTI also uses the polymer along with other salt or alkoxide additions to form matrix precursors for mullite, spinel and aluminum phosphate. Rockwell Science Center and ACT/Northwestern University have developed inorganic polymers for lanthanum phosphate ( $\text{LaPO}_4$ ), monazite, for use in fiber coatings and matrix processing.

#### 3.1.1.6.2 *Single oxide compositions*

A large body of literature, often containing step by step processing methods, is available for the formulation of single oxide sols such as silica ( $\text{SiO}_2$ ) (References 3.1.1.6.1(b) and (d)) or alumina ( $\text{Al}_2\text{O}_3$ ) (Reference 3.1.1.6.1(e)).

Silica-based ceramic matrix composites (CMCs) have been extensively studied for radome applications in conjunction with silica-based continuous reinforcement fibers. The temperature capability of these composites is limited to a maximum of 1799°F (982°C) to avoid excessive amorphous to crystalline phase transformation phenomena with associated microcracking of the matrix, and to avoid the annealing point of silica.

Alumina manufactured by the sol-gel process can be used to illustrate a classic example of single oxide alkoxide-based processing (Reference 3.1.1.6.1(e)). In this process an organometallic precursor is hydrolyzed, peptized with hydrochloric acid, and then fired at increasing temperatures to produce first gamma alumina, then alpha alumina. Alumina sol is currently being used to produce an interface coating on fibers for metal matrix composites and is a potential matrix precursor sol for ceramic composites requiring very high temperature matrix capability. Alumina sols derived from the Yoldas method have been investigated for use as matrices for silicon carbide whisker reinforced ceramic composites and silicon carbide fiber reinforced ceramic composites (References 3.1.1.6.2(a) and (b)).

#### 3.1.1.6.3 *Advantages and disadvantages of sol-gel processing*

*Advantages* -- One of the inherent advantages of sol-gel processing is very low temperature processing. Many of the currently available ceramic reinforcing fibers are life limited at temperatures greater than 1832°F (1000°C). A matrix processing method such as sol-gel that requires minimal exposure to tem-

temperatures greater than 1832°F (1000°C) limits thermal damage to the fibers and can reduce fiber/matrix interactions during processing. The sol-gel process can, therefore, extend the high temperature life of the final product by minimizing exposure to high temperatures during processing. Sol-gel matrix infiltration can be performed at very low temperatures (less than 572°F (300°C)) using either vacuum infiltration or autoclave molding techniques similar to those established for polymer matrix composites. The sol-gel technique allows processing of complex shapes, eliminates the need for high temperature tooling and, because the system does not have to be hot pressed, eliminates potential mechanical damage to the fibers in 3-D composites.

The tendency of the sol to wick along the individual filaments of the fiber tow bundles due to capillary action allows inexpensive, net-shape tooling to be used for complex 3-D shapes. Once the initial matrix infiltrations and low temperature firings of the woven structure have been accomplished, the CMC can be removed from the mold as it will no longer change shape. Subsequent infiltrations and firings including the final high temperature firing to sinter the ceramic material can, therefore, be performed without tooling. Further densification can be achieved with subsequent infiltrations using vacuum infiltration only, although the amount of porosity in the final product may be greater than desired. Higher densities can be achieved through the use of autoclave molding particularly during the initial cycles.

High-purity sol precursors are available, and purity can be easily maintained during processing. Standard ceramic matrix processing steps to reduce the precursor powder size such as ball milling or grinding are not required and, therefore, impurities are not introduced. Sintering aids to reduce the processing temperature are also not required as the processing temperatures are inherently low. Eliminating the need for a sintering aid such as boria ( $B_2O_3$ ) is advantageous because sintering aids usually reduce the overall temperature capability of the ceramic composite.

Complex, high purity oxide compositions not easily produced by other methods, including ternaries such as cordierite ( $2MgO-2Al_2O_3-5SiO_2$ ) and quaternary compositions such as Nasicon ( $Na_3Zr_2Si_2PO_{12}$ ), can be formulated with varying stoichiometries. Sol-gel processing is most often used for simpler matrix compositions such as alumina ( $Al_2O_3$ ), mullite ( $3Al_2O_3 \rightarrow 2SiO_2$ ), spinel ( $MgAl_2O_4$ ) or aluminum phosphate ( $AlPO_4$ ).

*Disadvantages* -- Some of the disadvantages associated with sol-gel processing of ceramic composites are based upon the developmental stage of this technology. Others are inherent to the technology. Although some form of alkoxide precursor chemical is typically available for most of the materials of interest, many are only available in small quantities and are often expensive. If the matrix sol formulation is performed on a laboratory scale using ultra-pure and expensive precursor chemicals, the product composite may be very expensive. Fortunately, sol-gel has now been extended to include inorganic polymeric solutions which generally use lower cost salt precursors rather than alkoxides. There are also now commercially available colloidal sols for alumina, silica, mullite, yttria and others.

Inherent shrinkage of the gelled sols due to the large volume of water and alcohols that must be removed can be severe and often results in a microcracked matrix. For alkoxide-based sols, typically excess water for hydrolysis is used to ensure complete hydrolysis reactions. Attempts to reduce the amount of water can result in poor mixing of the precursors, affecting the homogeneity of the sol and ultimately raising the processing temperature. The large amounts of solvent used also limit the yield of dried matrix obtained per infiltration; therefore, multiple infiltrations are often required in order to achieve the desired density.

The advantages and disadvantages of sol-gel processing of CMCs are summarized in Table 3.1.1.6.3.



**TABLE 3.1.1.6.3** *Advantages and disadvantages of sol-gel processing.*

<b>Advantages</b>	<b>Results</b>
Low Temperature Processing	Limits Fiber Damage Limits Fiber/Matrix Interaction Allows Inexpensive Tooling
Net-Shape Fabrication	2-D & 3-D Structures Possible Lowers Cost Minimizes Machining
High Purity	Lessens Reactivity Increases Temp. Capability
Controllable Compositions	Multicomponent Compositions Possible
<b>Disadvantages</b>	<b>Results</b>
High Shrinkage	Matrix Cracking
Low Yield	Multiple Infiltrations Required
High Precursor Costs	Current High Cost Limits Broader Interest
Acidic Gases Evolved During Pyrolysis	Can Cause Fiber Damage
Limited Shelf Life of Sols	Increases Matrix Costs

#### 3.1.1.6.4 *Drying control agents and fillers*

One of the most significant problems in sol-gel processing of continuous fiber reinforced ceramics is shrinkage during drying and sintering with subsequent cracking of the matrix. The addition of glycerol ( $C_3H_8O_3$ ) to an alumina sol enhanced the handleability of the dried product and resulted in less shrinkage cracking during the manufacture of silicon carbide whisker reinforced alumina composites (Reference 3.1.1.6.2(b)). In addition to glycerol, other drying control chemical agents (DCCAs) such as formamide ( $NH_2CHO$ ) have been used by Hench (References 3.1.1.6.4(a) and (b)) to produce large, transparent silica monoliths and may produce more manageable 2-D sol-gel prepreps by controlling drying shrinkage.

Ceramic powders can also be added to reduce the inherent shrinkage and cracking of the matrix as it loses solvent, to seed the sols for crystallization, and to maximize the extent of matrix densification per infiltration step. The addition of solid powder particles to the sol will often reduce the number and severity of drying cracks in the matrix and will increase the overall composite density. These fillers can substantially improve the yield per infiltration if care is taken to avoid prematurely closing off the surface-connected porosity.

Seeding sol-gel precursors can also result in significant changes to the final fired microstructure and may have implications regarding improving the strength and/or toughness of the final composite product. Additions of 4 wt% crystalline mullite powder to a colloidal mullite precursor gel resulted in a microstructure composed of equiaxed grains rather than the acicular (aspect ratio 3:1) grains typical in the unseeded sol microstructure (Reference 3.1.1.6.4(c)).

One approach being used by industry and academia to significantly reduce the number of processing steps, i.e., infiltrations, associated with sol-gel matrix processing is to pressure slip cast a matrix powder

into the fiber preform prior to sol-gel infiltrations. Fine diameter slurries are required to ensure that the particles fill the void space within the fiber tows. By careful control of the particle size distribution of the slurry, the packing density of the matrix particles within the preform void space can be optimized to limit the number of subsequent sol-gel infiltrations required to meet the targeted composite density.

#### *3.1.1.6.5 Sol-gel processing of 2-D structures*

Sol-gel processing of 2-D structures can be performed in a manner directly analogous to processing polymer matrix composites. Fabrics are either prepregged or a resin-transfer molding operation is employed. Once the sols are obtained either through laboratory scale processing or from a commercial source, polymeric binders may be added to enhance the ability of the sols to coat the fabrics, and fillers may be added to increase the final ceramic yield. The fabric may be dipped into the sol or a filled sol to form the prepreg. Once a prepreg is available, it may be cut, stacked, and autoclave cured similarly to the way that polymer composites are fabricated. The difference from polymer composites, which generally have a 100% yield, is that the ceramic composite preform has a low yield for the matrix phase, and hence additional infiltrations are often required to achieve high densities. However, some component systems such as General Electric's GEN IV<sup>TM</sup> use the formed CMC without additional infiltrations. Porosity levels of 20 to 25% are present in these materials.

#### *3.1.1.6.6 Sol-gel processing of 3-D structures*

One method of sol-gel processing of CMCs is to infiltrate a woven (or braided) 3-D preform manufactured from ceramic fibers. The advantage to utilizing 3-D weaves versus 2-D lay-ups for ceramic composites is the structural reinforcement provided by the fiber in the through-thickness direction of the 3-D weave.

In order to enhance densification of the 3-D composites, traditional polymer matrix composite fabrication techniques, such as vacuum bagging followed by autoclave molding can be used. Currently fiber preforms are infiltrated by either using vacuum infiltration techniques or by autoclave molding. The sol is prepared or obtained commercially and poured into a container in a vacuum chamber. The 3-D preform is completely immersed in the sol while a low pressure vacuum is applied to remove air bubbles and assist movement of the sol into the interior of the weave. The sol is then gelled using either heat (302°F (150°C)) or catalyst (if appropriate) and the infiltration step is repeated until the desired density is achieved. Low temperature heat treatments (212-392°F (100 - 200°C)) of the gelled product after each infiltration will remove the physically adsorbed water and alcohols and slightly higher temperatures (572-752°F (300 - 400°C)) will drive off any residual organics. Further densification is often desired for enhanced mechanical properties and can be achieved with low pressure (50 to 100 psi) autoclave molding. Autoclave molding has the added advantage of allowing close tolerances to be achieved, which in turn reduces any subsequent machining.

Once the composite preform is rigidized, typically after two or three infiltration cycles, the remainder of the infiltration cycles can be performed without tooling. A freestanding firing cycle is employed after the desired density is achieved to produce a crystalline ceramic matrix composite. The low viscosity sol easily wicks along the filaments into the interior of the 3-D preforms with or without vacuum pressure being applied. Each infiltration produces a thin film coating on the filaments with subsequent infiltrations producing thicker filament coatings until the tow bundles are completely infiltrated. Eventually the surface porosity does begin to close but typically not before the desired final density is achieved.

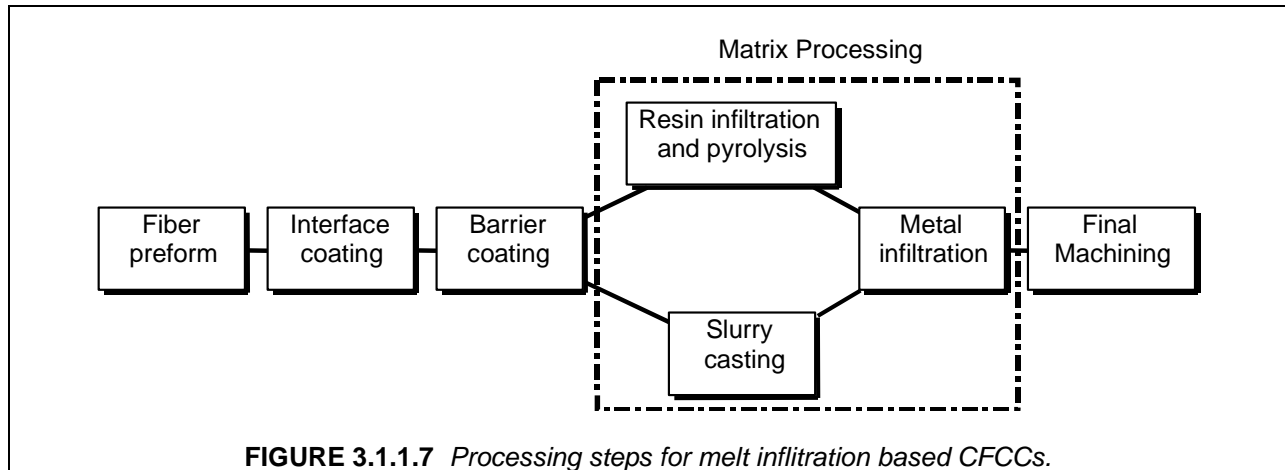
Techniques have been demonstrated to use slurry coating of 3-D preforms as a means for achieving substantial matrix densification in a single processing step. McDermott Technology, Inc. utilizes 3-D cylindrical preforms woven by Techniweave Inc. Special tooling compresses the wall of the cylinder to achieve a higher fiber loading. A slurry is pressure cast into the preform to build up a sediment of the matrix powder through the thickness of the preform. Prior to drying the part, a sol-gel matrix precursor is added which provides strength to the infiltrated preform prior to an initial firing step. Subsequent sol-gel infiltrations are then performed if additional matrix densification is desired. This processing approach works well for flat or cylindrical preforms, but is difficult with stiffened structures.

### 3.1.1.6.7 Summary

Sol-gel processing of ceramic matrix composites is a low temperature alternative to conventional high temperature ceramic processing techniques. It offers several significant processing advantages including net-shape fabrication, adaptability to fully woven structures, easy scalability and simple, inexpensive tooling. Sol-gel processing is a very attractive means of producing large, complex shapes but widespread use of this technology is currently hindered by low mechanical properties. Improvement of the mechanical properties requires research in the following areas: control of matrix cracking during drying, higher temperature matrices, matrix densification, fiber/matrix interface control, and ceramic fibers that maintain high strength at temperature.

### 3.1.1.7 Melt infiltration

Melt infiltration is a matrix fabrication process which uses a molten metal to infiltrate the residual porosity in fiber preforms. For this process the fiber reinforcement is typically coated with an interface and protective layer prior to infiltration. The matrix material resulting from this process is typically a combination of the infiltrated metal in solid form and some secondary components. The key process steps necessary for fabrication of a Melt Infiltrated CFCC are shown in Figure 3.1.1.7.



Preparation of the fiber reinforcement consists of forming it into the desired shape and applying an interface coating (BN, carbon, etc.) and barrier coating (SiC, Si<sub>3</sub>N<sub>4</sub>, etc.). Typically these coatings are applied using CVD techniques. The interface coating provides for load transition between the matrix and fiber. The barrier coating is applied to protect the interface and fibers from the high temperature molten metal during the infiltration.

The initial matrix processing step involves one of two methods for adding components to the pores of the coated preform. These two methods are:

1. Resin infiltration, pyrolysis, and reaction
2. Slurry casting

#### 3.1.1.7.1 Resin infiltration, pyrolysis, and reaction

In this method the coated preform is infiltrated with a liquid acrylic resin which is pyrolyzed leaving a supply of reactant components (most typically carbon) within the porosity of the preform. During the metal infiltration process, these components react with the molten metal and form the secondary components of the matrix.

### 3.1.1.7.2 Slurry casting

In this method the preform is subjected to a slurry casting procedure to place the secondary matrix components directly into the pores of the preform. These components survive the metal infiltration process and form an integral part of the final matrix.

The final matrix processing step is metal infiltration. In this step the prepared preform is heated under vacuum to a temperature above the melting point of the chosen metal. A supply of this metal is placed in contact with the preform such that it wicks into its pores. After a short duration at temperature the preform is cooled and the metal solidifies leaving a rigid composite structure. Table 3.1.1.7.2 shows typical systems and representative properties.

In comparison to other forms of matrix densification the melt infiltration technique has many benefits. They include:

Less open porosity	Dense matrix provides:
Greater thermal conductivity	Superior fiber/interface protection
Less oxidation/environmental degradation	Higher proportional limit
Potentially shorter processing times	

**TABLE 3.1.1.7.2** *Typical systems and representative properties.*

Matrix	Fiber/ Reinforcement	Manufacturers	
Si - SiC	Hi-Nicalon™, Sylramic™, Carbon	Honeywell Advanced Composites Inc., B.F. Goodrich, General Electric....	
		Ambient	2192°F (1200°C)
Fiber: Hi Nicalon™	Ultimate Tensile Stress	52.7 ksi (363 MPa)	38.1 ksi (263 MPa)
Interface: Boron Nitride	Ultimate Tensile Strain	0.75 %	0.55 %
Matrix: Melt Infiltrated Si-SiC via slurry casting	Proportional Limit	17.6 ksi (121 MPa)	207 ksi (143 MPa)
	Tensile Modulus	29.2 Msi (201 GPa)	35.1 Msi (242 GPa)

### 3.1.1.8 Reaction processing

Reaction processing or reactive forming refers to techniques where the matrix of the composite is formed from several phases that react during processing to form a new phase. Generally, the preforms consist of reinforcements surrounded by a dense compact which is one (or more) of the matrix precursors. The compact is then reacted with a gas, a molten material, or itself. These techniques utilize reactants which can be manipulated as slurries or powders, and have low to moderate melting temperatures. These features are advantageous over the traditional ceramic processing methods which involve very high sintering temperatures and shrinkage, or are hampered by the fact that many ceramics sublime or decompose instead of melting. Other advantages of reaction processing methods include the ability to form complex and near-net shapes, to densify in a single step, and, in some cases, to achieve zero or low porosity.

Important factors to consider in the reaction processing of CMCs include the final density and whether or not the reinforcements are damaged. A dense matrix results from a volume increase upon reaction, so candidate reactions usually result in a product with a density lower than that of the starting materials. Reinforcing fibers can be damaged by exposure to elevated temperatures or by the forming reaction. To

avoid damaging the fibers, consider systems with sufficiently low reaction temperatures and low chemical affinity for the fiber. In cases where the reactants might chemically attack the fibers, protective interface coatings may be applied.

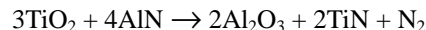
Two reaction processes, the DIMOX™ process and melt infiltration, were discussed earlier in this chapter. Other examples of reaction processes follow:

*Reaction Bonded Silicon Nitride.* Reaction bonded silicon nitride (RBSN) is fabricated by forming a compact of silicon particles in the desired shape and exposing this porous compact to a nitrogen atmosphere at temperatures starting at about 2192°F (1200°C) and ending at about 2552°F (1400°C). The silicon reacts with the nitrogen to form silicon nitride. The reaction involves about 60% weight gain, which is accommodated by growth of the silicon nitride into the pores. This results in an increase in density to about 80-85% of theoretical with less than 1% change in external dimensions. The silicon-nitrogen reaction is exothermic, so the reaction is carefully controlled to avoid having the temperature exceed the melting temperature of silicon. Depending on the size of the part being nitrided, the reaction process can take from a few days to over a week (Reference 3.1.1.8(a)). Due to potentially long exposures at elevated temperatures, reinforcing fibers can be damaged when using this process for CMC fabrication. Monofilament fibers such as SCS-6 CVD SiC have shown stability during nitridation, but polymer derived multifilament fibers can degrade. Nitride bonded silicon carbide (discussed next) offers a solution to the fiber degradation.

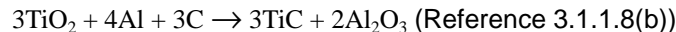
*Nitride Bonded SiC.* Nitride bonding uses the reaction of nitrogen with silicon to form a bond phase between SiC particles rather than the composite matrix. The reduced concentration of silicon allows nitridation in a much shorter time than for RBSN, which allows for a wider selection of fiber materials. Textron Specialty Materials has successfully fabricated nitride bonded SiC (NB-SiC) composites with SCS-6 CVD monofilaments, Tyranno SiC-based multifilament fiber, and carbon fibers (Reference 3.1.1.8(a)).

*Reactive Sintering and Combustion Synthesis.* The term reactive sintering implies that two or more constituents in a compact react during sintering to form a new phase or phases. The reaction is normally exothermic and can contribute to an enhancement of sintering. In some cases the reaction is so exothermic that it can generate sufficient heat to cause self-sintering without external heating except that required for initiating the reaction. This is the basis of combustion synthesis, which if properly controlled, can produce a relatively dense compact of the synthesized reaction product. Alternatively, pressure can be applied immediately after the reaction thus utilizing the heat of reaction in further consolidation. Two examples of composites produced are:

- Reaction sintering:



- Combustion synthesis:



*Cement Matrix Composites.* A variety of ceramic compositions can be achieved through cementitious bonding. These bonds can form at low temperature and typically involve hydration reactions (as Portland cement or plaster) or other chemical reactions such as an oxide with phosphoric acid. One of the earliest successful ceramic matrix composites consisted of glass fiber reinforced cement (GFRC). Another example is cement reinforced with 1-3% discontinuous carbon fibers. The fibers alter the fracture behavior and instill high toughness and improved reliability (Reference 3.1.1.8(a)).

### 3.1.1.9 Fibrous monoliths (FMs)

#### 3.1.1.9.1 Introduction

In general, fibrous monoliths (FM's) are a new class of structural ceramics. They have mechanical properties similar to CFCCs, including very high fracture energies, damage tolerance, and graceful fail-

ure. But since they are monolithic ceramics, fibrous monoliths can be manufactured by conventional powder processing techniques using inexpensive raw materials. This combination of high performance and low cost is a breakthrough which could enable wider application of ceramics in energy and defense related applications. FMs are sintered (or hot pressed) monolithic ceramics having a distinct fibrous texture. The two phases that make up the macroarchitecture of an FM are a primary phase in the form of elongated polycrystalline cells, or fibers, separated by a thin secondary phase in the form of cell boundaries. Typical volume fractions of the two phases are 80 to 95% for the primary (cell) phase, and 5 to 20% for the interpenetrating (cell boundary) phase. The primary (cell) phase is typically a structural ceramic, such as  $\text{Si}_3\text{N}_4$ ,  $\text{SiC}$ ,  $\text{ZrB}_2$ ,  $\text{HfB}_2$ ,  $\text{HfC}$ ,  $\text{ZrO}_2$ , or  $\text{Al}_2\text{O}_3$ , while the interpenetrating (cell boundary) phase is typically either a ductile metal, such as Ni, Ni-Cr, Nb, or a weakly bonded low shear strength material, such as graphite or hexagonal BN. Table 3.1.1.9.1 lists many of the FM material systems that have been developed for various applications.

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**TABLE 3.1.1.9.1** *List of FM material systems that have been fabricated for a host of applications.*

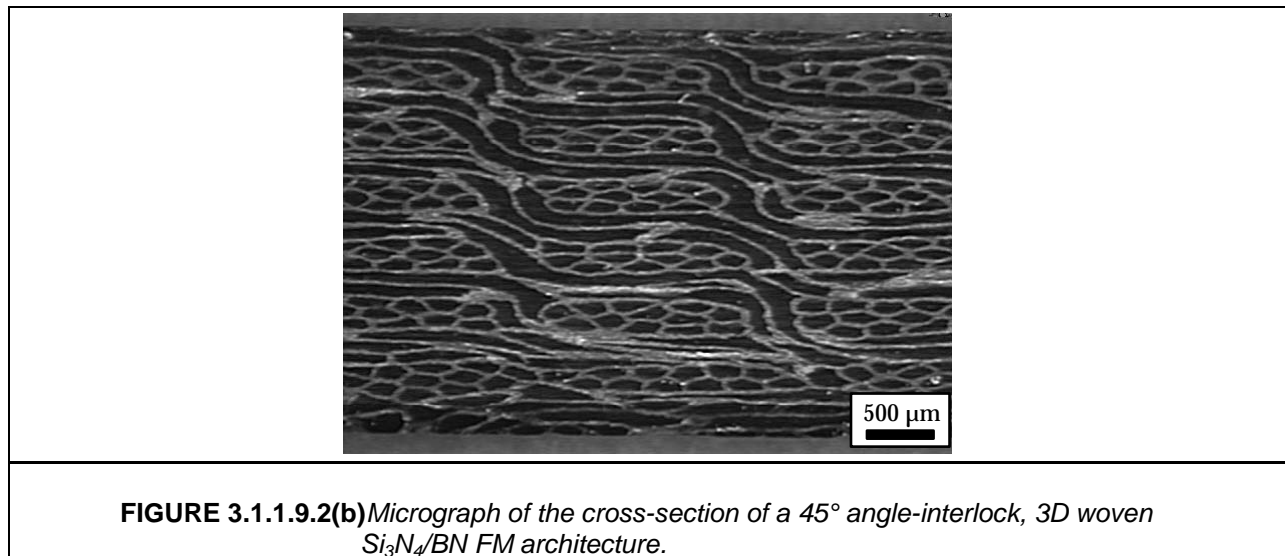
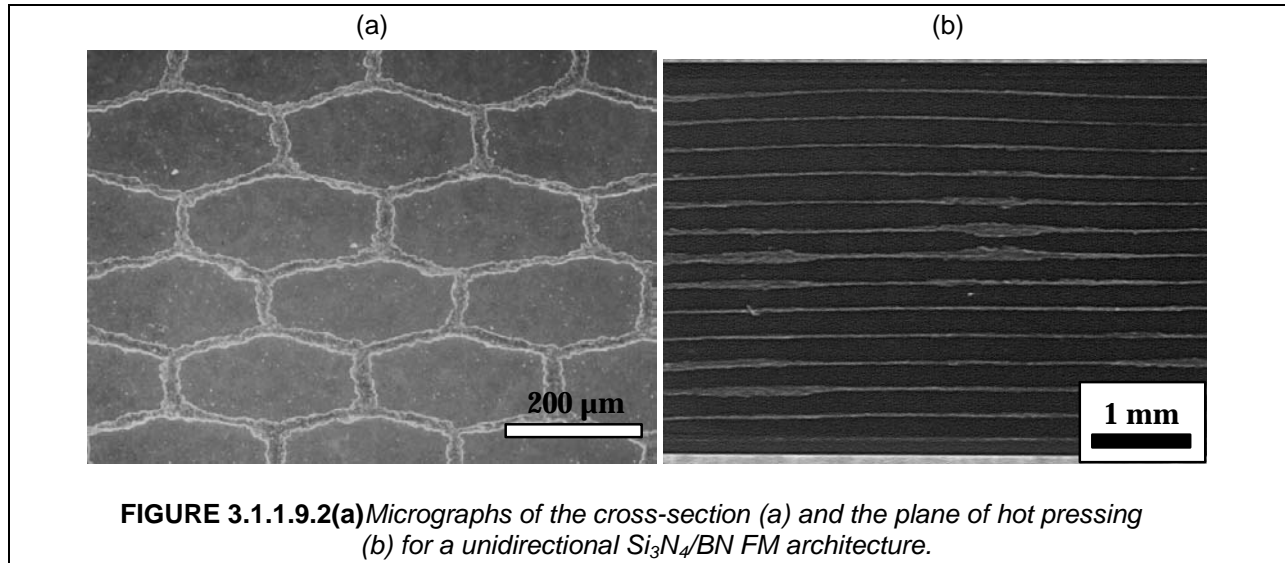
<ul style="list-style-type: none"> <li>• <b><u>Nitrides</u></b> <math>\text{Si}_3\text{N}_4/\text{BN}</math></li> </ul>	<ul style="list-style-type: none"> <li>• <b><u>Diborides</u></b> <math>\text{ZrB}_2/\text{BN}</math> <math>\text{HfB}_2/\text{BN}</math></li> </ul>	<ul style="list-style-type: none"> <li>• <b><u>Cermet-based FMs:</u></b> <math>\text{WC}/\text{CO}</math></li> </ul>
<ul style="list-style-type: none"> <li>• <b><u>Carbides</u></b> <math>\text{SiC}/\text{BN}</math> <math>\text{SiC}/\text{graphite}</math> <math>\text{SiC}-\text{AlN}/\text{BN}</math> <math>\text{HfC}/\text{W}-3.6\text{w}\%\text{Re}</math> <math>\text{Diamond}/\text{WC}</math></li> </ul>	<ul style="list-style-type: none"> <li>• <b><u>Oxides</u></b> <math>\text{Al}_2\text{O}_3/\text{graphite}</math> <math>\text{Al}_2\text{O}_3/\text{Ni}</math> <math>\text{Al}_2\text{O}_3/\text{Ni}-20\text{CR}</math> <math>\text{ZrO}_2(\text{PSZ})/\text{Ni}</math></li> </ul>	<ul style="list-style-type: none"> <li>• <b><u>Trilayer FMs:</u></b> <math>\text{Si}_3\text{N}_4/\text{BN}/\text{Si}_3\text{N}_4</math> <math>\text{ZrO}_2(\text{PSZ})/\text{Al}_2\text{O}_3/\text{Ni}</math></li> </ul>

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The basic FM architecture is fabricated using a simple process for converting ordinary raw powder into a “green” filament consisting of the powder and a thermoplastic polymer binder. These filaments can be compacted in the “green” state to create a fabric of polycrystalline cells after sintering. The secondary phase for the cell boundary is introduced during the fabrication process, providing a bi-component or even multi-component filament during melt spinning. The process is widely applicable, allowing cell/cell boundary filaments to be made from any thermodynamically compatible set of materials available as sinterable powders. The scale of the microstructure is determined by the green fiber diameter (for cell size) and coating thickness (for cell boundaries). Once the green composite filament is fabricated, it can be wound or braided into the shape of the desired component using any conventional composite architecture (e.g., uniaxial lay-up, biaxial lay-up, woven fabric, etc.). After forming the desired component, the binder is removed via heating, and final consolidation of the body typically occurs through hot-pressing.

### 3.1.1.9.2 Macrostructure of fibrous monoliths

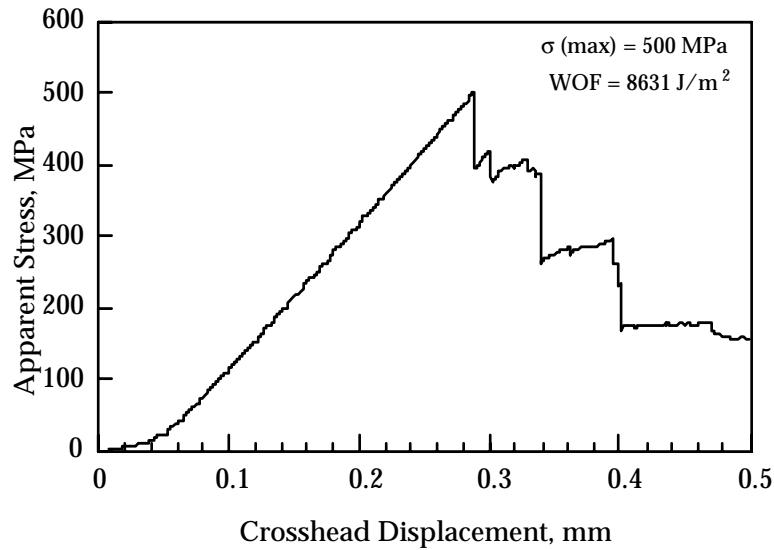
Figure 3.1.1.9.2(a) shows a typical polished cross-section and side surface of a uniaxially aligned  $\text{Si}_3\text{N}_4/\text{BN}$  FM architecture. The polycrystalline  $\text{Si}_3\text{N}_4$  cells appear in dark contrast, and the continuous BN cell boundaries appear in bright contrast. Figure 3.1.1.9.2(b) shows the cross-section of a 45° angle-interlock, 3D woven  $\text{Si}_3\text{N}_4/\text{BN}$  FM architecture.



### 3.1.1.9.3 Mechanical properties of fibrous monoliths

FM systems have been fabricated achieving flexural strengths as high as 102 ksi (700 MPa), fracture energies approaching  $6.1 \times 10^{-3}$  Btu/in<sup>2</sup>s (10,000 J/m<sup>2</sup>s), and materials with measured use temperatures of 4040°F (2500 K). Figure 3.1.1.9.3 is a typical load-deflection curve for a unidirectional  $\text{Si}_3\text{N}_4/\text{BN}$  weak interface FM tested in four-point flexure at room temperature, showing a peak apparent flexural strength of 73 ksi (500 MPa) with significant load-bearing capacity after the first fracture event occurs. It is the area under the curve that is used to calculate the work of fracture (WoF) for this material,  $\sim 5.3 \times 10^{-3}$  Btu/in<sup>2</sup>s ( $\sim 8,600$  J/m<sup>2</sup>s).

Fibrous monolithic ceramics trade a portion of the inherent flexural strength of the base material ( $\text{Si}_3\text{N}_4$  in this case) for a one to two orders of magnitude increase in fracture toughness. The result is a material which fails gracefully as the crack energy is consumed by delamination and sliding at the cell-boundaries. Table 3.1.1.9.3 shows the typical room temperature flexural properties of  $\text{Si}_3\text{N}_4/\text{BN}$  and Diboride/BN FMs in several architectures.



**FIGURE 3.1.1.9.3** Load-deflection curve for a  $\text{Si}_3\text{N}_4/\text{BN}$  fibrous monolith tested in four-point flexure at room temperature.

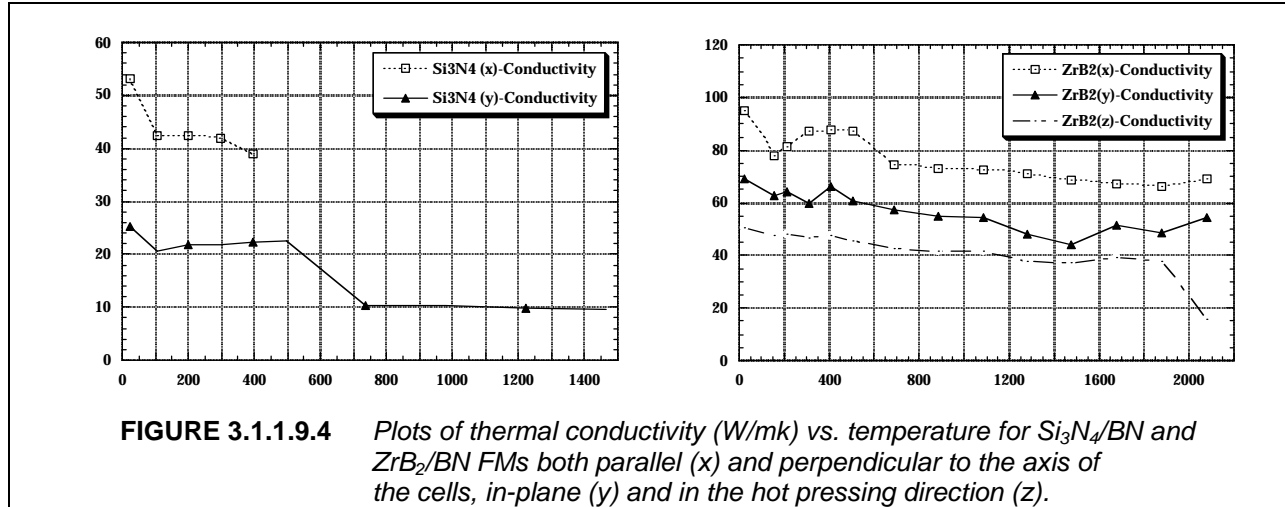
#### 3.1.1.9.4 Thermal properties of fibrous monoliths

The variation in thermal conductivity with temperature, as well as the degree of thermal anisotropy in these FM systems is shown in Figure 3.1.1.9.4. Thermal conductivity values were obtained through thermal diffusivity measurements on multifilament coextruded  $\text{Si}_3\text{N}_4/\text{BN}$  and diboride/BN FMs using a laser flash technique. The thermal diffusivity was measured both parallel and perpendicular to the fibrous architecture on uniaxial coupons containing nominally 85 vol.% cell material and 15 vol.% cell boundary material.

**TABLE 3.1.1.9.3** Typical room temperature mechanical properties for  $\text{Si}_3\text{N}_4/\text{BN}$  and Diboride/BN FMs tested in four-point bending.

FM – Architecture	E, Msi	$\sigma$ , ksi	WoF, Btu/in <sup>2</sup> s
$\text{Si}_3\text{N}_4/\text{BN}$ – uniaxial	39.7±0.7	66.1±19.9	$4.96 \times 10^{-3} \pm 5.6 \times 10^{-4}$
$\text{Si}_3\text{N}_4/\text{BN}$ - 0°/90°	28.7±0.6	48.4±5.1	$3.91 \times 10^{-3} \pm 5.6 \times 10^{-4}$
$\text{Si}_3\text{N}_4/\text{BN}$ - 0°/±45°/90°	30.3±0.6	38.0±2.5	$1.99 \times 10^{-3} \pm 2.14 \times 10^{-4}$
$\text{ZrB}_2/\text{BN}$ – uniaxial	53.5±1	46.4±5.9	$7.18 \times 10^{-4} \pm 9.73 \times 10^{-5}$
$\text{ZrB}_2/\text{BN}$ - 0°/90°	48.9±1.5	19.6±2.8	$1.98 \times 10^{-4} \pm 2.69 \times 10^{-5}$
$\text{HfB}_2/\text{BN}$ – uniaxial	55.0±3.5	42.4±5.5	$6.56 \times 10^{-4} \pm 7.89 \times 10^{-5}$
$\text{HfB}_2/\text{BN}$ - 0°/90°	46.9±1	24.2±2.8	$2.16 \times 10^{-4} \pm 3.37 \times 10^{-5}$





### 3.1.1.9.5 Applications for fibrous monoliths

Applications for FMs are widespread and have recently included structures such as flat plates, solid hot gas containment tubes, radiant burner panels, rocket nozzles and rocket propulsion components, ballistic armor panels for aircraft and ground vehicles, and drilling/cutting tool components, which can all be readily formed from the green material.

Applied development work on FMs, being performed at Advanced Ceramics Research, Inc. in Tucson, Arizona, has mainly focused on the development of  $\text{Si}_3\text{N}_4/\text{BN}$  FMs for intermediate temperature turbine engine applications as well as  $\text{ZrB}_2$ -based/BN,  $\text{HfB}_2$ -based/BN, and  $\text{HfC}/\text{W-Re}$  FMs for both rocket and solar thermal propulsion applications. Recent development efforts have also led to the development of  $\text{Diamond}/\text{WC}^{\text{TM}}$  FMs for use as tool bit inserts for the petroleum drilling industry. Improvements in the mechanical behavior of these materials has led to significant product improvements.

### 3.1.1.10 Hybrid systems

Reserved for future use.

## 3.2 FIBER/REINFORCEMENT SYSTEMS AND TECHNOLOGY

### 3.2.1 Introduction – the role and function of reinforcements in CMCs

The ceramic fibers in ceramic matrix composites are the direct load-bearing components in the composite. They act as the key structural factors in providing the high strength, the modulus, and the damage tolerance within the system.

Many different types of ceramic fibers have been produced with a wide range of compositions, geometries, and properties. The following sections describe the currently available ceramic fiber types.

### 3.2.2 Continuous fibers

#### 3.2.2.1 Oxide fibers

Commercial polycrystalline oxide fibers used for the reinforcement of ceramic composites are produced by spinning and pyrolyzing chemically-derived precursors. This approach is also commonly called

sol/gel processing. The use of chemical processing allows the preparation of fibers with high alumina contents and, therefore, superior high temperature properties compared with fibers produced by spinning of molten glasses from silica-based melts, which have a maximum of 55%  $\text{Al}_2\text{O}_3$ . A wide variety of precursors and processing techniques exist for both oxide and non-oxide fibers; these have been used to control fiber microstructural development and a variety of fiber properties, such as modulus, density, dielectric constant and tensile strength.

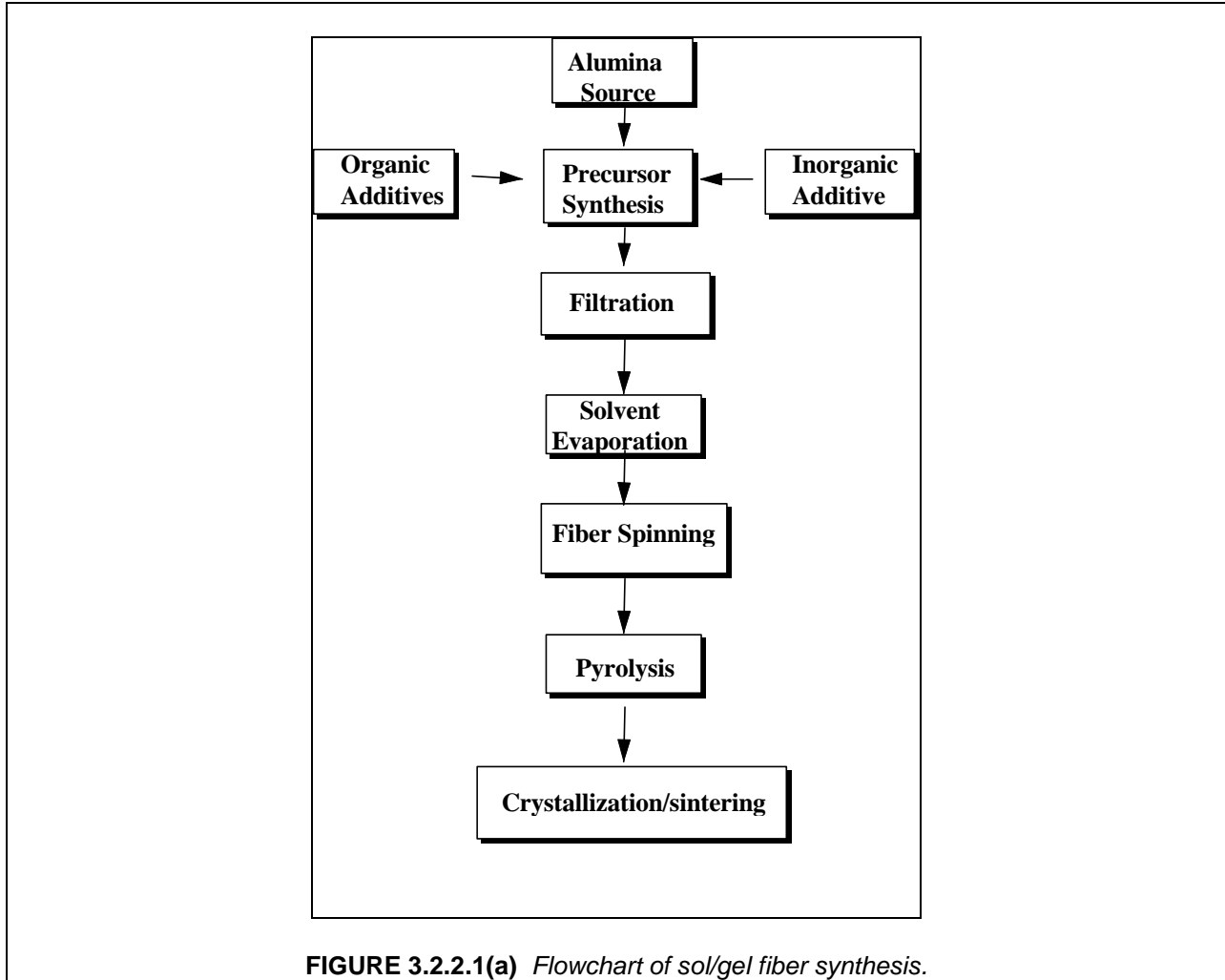
Oxide fibers are typically available as continuous, multifilament tows or rovings. Fiber tows, which typically consist of 500 - 1000 filaments each having a diameter of 10 - 15  $\mu\text{m}$ , are flexible and easily handled, which allows them to be woven into fabrics and used for the fabrication of composites with complex shapes. One key characteristic of oxide and other reinforcement fibers is their ultra-fine or even nanoscale microstructure; a grain size below 0.5  $\mu\text{m}$  is optimal for high strength. The small grain size has the disadvantage of increasing the rate of high temperature creep. However, progress in fiber processing over the last decade has enabled the preparation of both oxide and non-oxide fibers with significantly improved high temperature creep properties.

All commercial polycrystalline oxide fibers used in CFCCs are based on alumina. High alumina content provides a number of advantages for a reinforcing fiber, including increased chemical stability, high melting point, high modulus, and good strength up to 2192°F (1200°C). A number of alumina precursors suitable for forming fibers are available. The aqueous chemistry of aluminum allows for the formation of viscous basic aluminum salt solutions which can be made into fibers by dry-spinning. More recently, polymeric aluminoxane precursors have also been used as a route to commercial alumina-based fibers. Silica is used as an additive for several reasons. Most also contain  $\text{SiO}_2$ , in amounts from 0% - 24%. Silica stabilizes alumina-based fibers with respect to detrimental crystallization to  $\alpha\text{-Al}_2\text{O}_3$  and mullite, which can lead to the formation of coarse-grained, porous microstructure and, therefore, low fiber strength.

Figure 3.2.2.1(a) shows a flowchart for sol/gel fiber synthesis. The fiber precursor is synthesized from a mixture of an alumina precursor, precursors of other inorganic additives, such as a silica, and organic additives. The synthesis method provides a chemistry which allows the preparation of a high viscosity liquid suitable for fiber spinning. The spinning solution or spin dope must be stable with respect to both crystallization/precipitation of insoluble complexes and to rapid increases in viscosity or gelation resulting from progressive cross-linking of precursor species. Common alumina precursors of commercial fibers include aluminum chlorohydrate (Almax™ Alumina Fibers), aluminum acetate (Nextel™ series of fibers), and aluminoxane polymers (Sumitomo™ Altex).  $\text{SiO}_2$  precursors used as a method of introducing  $\text{SiO}_2$  into fiber compositions include colloidal silica sols and partially hydrolyzed silicon alkoxides and polysiloxanes. Other inorganic modifiers to oxide fiber compositions can be added using soluble salts (e.g., nitrates).

Figure 3.2.2.1(b) shows a schematic of a typical commercial fiber-making process. The spin dope, which typically has a viscosity of 100-1000 Pa-s is pumped through a multiple orifice spinneret to form 400-1000 filaments or more simultaneously. Spinning is performed in a spinning tower using carefully controlled humidity, temperature, and air flow conditions. The fiber becomes rigid as solvent is evaporated. Fiber diameter can be controlled by varying the volumetric pumping rate relative to the speed of the draw wheels.

After spinning, the green fibers are conveyed into a furnace for heat treatment. Pyrolysis, or the conversion of the chemical precursor into ceramic, involves the loss of 50-80% of the initial weight of the green fiber. Volatile components must diffuse out of the fiber during this stage. Thus, pyrolysis must be performed with extreme care to gently decompose the green fiber to the oxide form without forming defects or flaws which reduce fiber strength. Pyrolysis is complete by 1472°F (800°C); above 1472°F (800°C), heat treatment causes the fiber to crystallize to alumina or other compounds, depending on the composition and precursors chosen. Sintering can occur before or after crystallization.



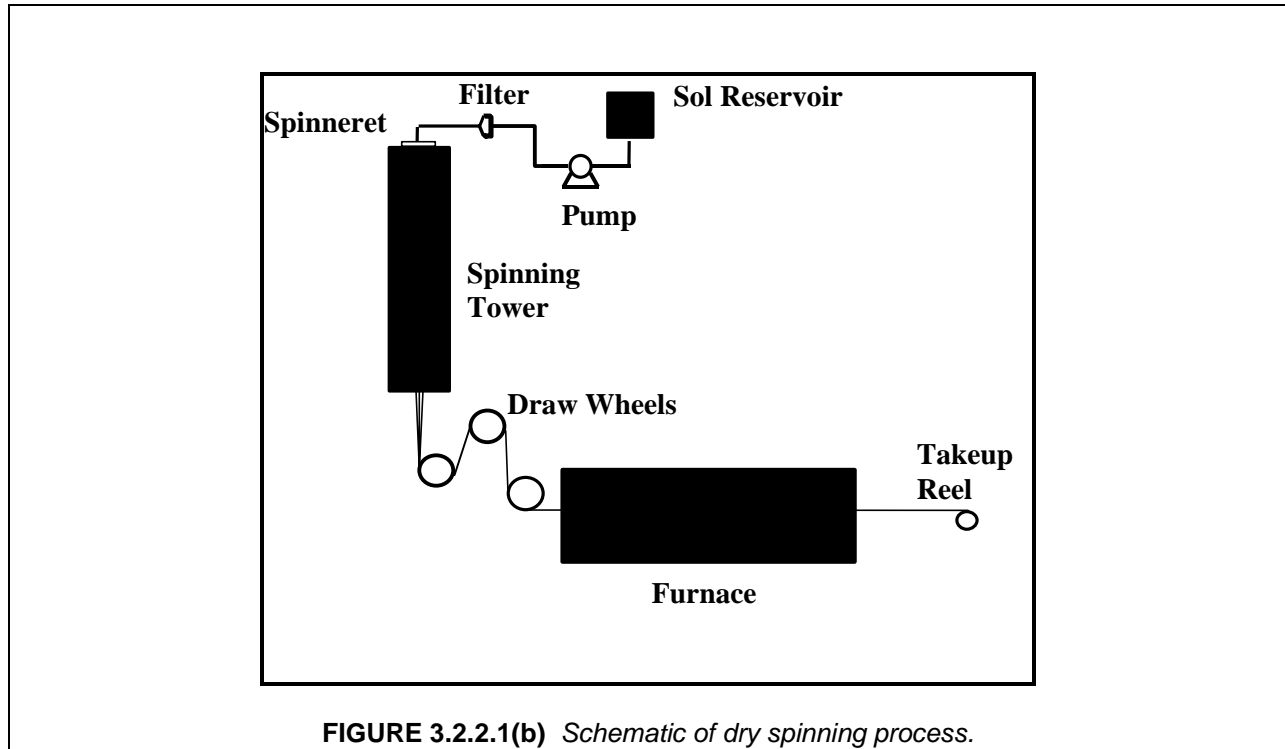
**FIGURE 3.2.2.1(a)** Flowchart of sol/gel fiber synthesis.

The goal of heat treatment is to develop a ceramic microstructure with both good strength and good high temperature properties (i.e., creep, creep rupture, and resistance to thermal degradation via grain growth). To achieve high fiber strength at room temperature, a small grain size (0.5  $\mu\text{m}$ ) is required. The Griffith equation,  $\sigma = K_{Ic} \sqrt{\pi c}$ , indicates that for  $\text{Al}_2\text{O}_3$ , ( $K_{Ic} = 4$ ), a strength of 3 GPa can only be achieved if the flaw size  $c$  is 0.6  $\mu\text{m}$ . Thus, not only should process-related flaws be kept small, but grain size should also not exceed this limit.

Many commercial oxide fibers have a major phase of transition alumina stabilized by amorphous silica (e.g., Nextel™ 550, Sumitomo Altex). Transition aluminas, such as  $\eta\text{-Al}_2\text{O}_3$  (cubic) or  $\gamma\text{-Al}_2\text{O}_3$  (tetragonal), are a series of alumina spinels (differentiated by varying degrees of ordering on the cation lattice) which form during heat treatment of alumina precursors at 1472 - 1652°F (800 – 900°C). Transition aluminas have small grain size, typically 100 nm or less. Fibers of this type are stable up to about 1200°C. Above that temperature, the transition alumina transforms to  $\alpha\text{-Al}_2\text{O}_3$ , or, in the presence of silica, to mullite. Typically, fiber strength is reduced during this crystallization due to the growth of large crystals, which act as flaws.

A variant of this type of fiber is Nextel 312, which contains 14%  $\text{B}_2\text{O}_3$  in addition to alumina and silica in the 3:2 atomic ratio of mullite. Instead of  $\gamma\text{-Al}_2\text{O}_3$ , Nextel 312 crystallizes to  $9\text{Al}_2\text{O}_3\cdot 2\text{B}_2\text{O}_3$ , which gradually transforms to mullite via exchange of Si and B ions in the crystal lattice during further heat treatment above 1000°C. The high  $\text{B}_2\text{O}_3$  content provides several advantages, including low density, low thermal

expansion and low elastic modulus (and, therefore, higher flexibility and strain to failure) than the other fibers. However, the  $B_2O_3$  degrades the high temperature performance of the fiber (e.g., creep), and also makes the fiber more reactive with potential oxide matrices.



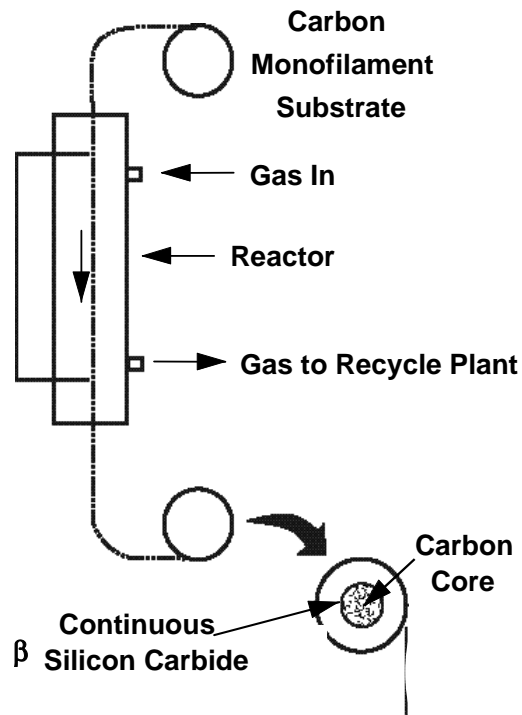
**FIGURE 3.2.2.1(b)** Schematic of dry spinning process.

Several oxide fibers contain  $\alpha\text{-Al}_2\text{O}_3$  as a major phase. Two commercial fibers, Nextel 610 and Mitsui Almax, are >99%  $\text{Al}_2\text{O}_3$  in the alpha phase. Thus, these fibers have superior chemical and thermal stability than the silica-containing fibers and are, therefore, less reactive with potential oxide matrices during fabrication and are more stable in corrosive service environments. Disadvantages of the high alumina content are higher density, and higher elastic modulus, which lowers strain-to-failure and flexibility during handling.  $\alpha\text{-Al}_2\text{O}_3$  also has intrinsically high fracture resistance, allowing higher strength fibers to be produced. For instance, Nextel™ 610 fibers have a tensile strength of 440 ksi (3.0 GPa), which is 50% higher than other commercially available fibers. Fiber FP, another polycrystalline  $\alpha\text{-Al}_2\text{O}_3$  fiber, which has been characterized extensively in the literature, is no longer commercially produced.

Another fully crystalline fiber is Nextel™ 720, which consists of a two-phase mixture of  $\alpha\text{-Al}_2\text{O}_3$  and mullite. This fiber has superior creep performance compared with other oxide fibers due to its high content of mullite, a highly creep-resistant compound, and to its unique crystalline structure or interpenetrating phases which reduces deformation by grain boundary sliding. Sumitomo Altex, which has almost identical chemical composition, has inferior creep resistance because its microstructure consists of nano-scale  $\gamma\text{-Al}_2\text{O}_3$  and amorphous silica. Deformation rate under stress is higher due to the presence of the amorphous phase.

### 3.2.2.2 SiC monofilaments

Continuous silicon carbide monofilaments are fabricated by chemical vapor deposition (CVD) of beta silicon carbide on a continuous carbon monofilament substrate. This process, shown schematically in Figure 3.2.2.2(a), has been used to make filaments with both graded and layered structures.



**FIGURE 3.2.2.2(a)** Production of SiC monofilaments on a carbon core substrate using chemical vapor deposition (CVD).

These types of fiber, fabricated by Textron and designated as “SCS”, consist of stoichiometric SiC with a columnar grain structure which radiates outward from the carbon core (Figure 3.2.2.2(b)). Carbon-rich surface coatings provide a toughness-enhancing parting layer in composites having a brittle matrix, such as silicon nitride or silicon carbide.



**FIGURE 3.2.2.2(b)** Micrograph of cross section of SCS fiber.

In contrast to other processes for producing SiC fibers, chemical vapor deposition has the advantage of rapid high temperature layer-by-layer formation of the SiC microstructure without the introduction of deleterious oxide phases. However, the mechanisms for SiC formation from silicon and carbon-containing chemical precursors are very complicated and quite sensitive to a number of parameters including reactant gas species, carrier and/or reducing gas, deposition pressure, temperature, gas phase concentrations, flow rate, etc.

A cross section of this type of CVD SiC fiber is shown in Figure 3.2.2.2(b). SCS fiber diameters that are commercially available as reinforcements for ceramics are 142 and 79 microns and are designated as SCS-6 and SCS-9A, respectively.

A third fiber, SCS-ULTRA™, was recently developed to achieve higher strength and is available in a 142 micron diameter. The strength of the SCS-ULTRA fiber is substantially higher than any other commercial reinforcement fiber, 957 ksi (6.6 GPa) (Table 3.2.2.2).

**TABLE 3.2.2.2 CVD SiC filament properties.**

This is information obtained from the Textron WEB site (26<sup>th</sup> February 2001) (<http://www.systems.textron.com/sicfiber.htm>) Textron Fiber Properties

<b>CVD Silicon Carbide Fibers</b> Property at Room Temperature	SCS-Ultra	SCS-6	SCS-9A
Nominal Composition			
Fiber Diameter (microns)	140	140	79
Density (g/cm <sup>3</sup> )	NA	3.0	2.8
Tensile Strength ksi (MPa)	400+ (6210+)	500 (3450)	500 (3450)
Young's Modulus Msi (GPa)	60 (414)	60 (400)	44 (307)
Coef. of Linear Thermal Expansion (ppm/K)	NA	2.3 at 73°F (23°C)	4.3 at 73°F (23°C)

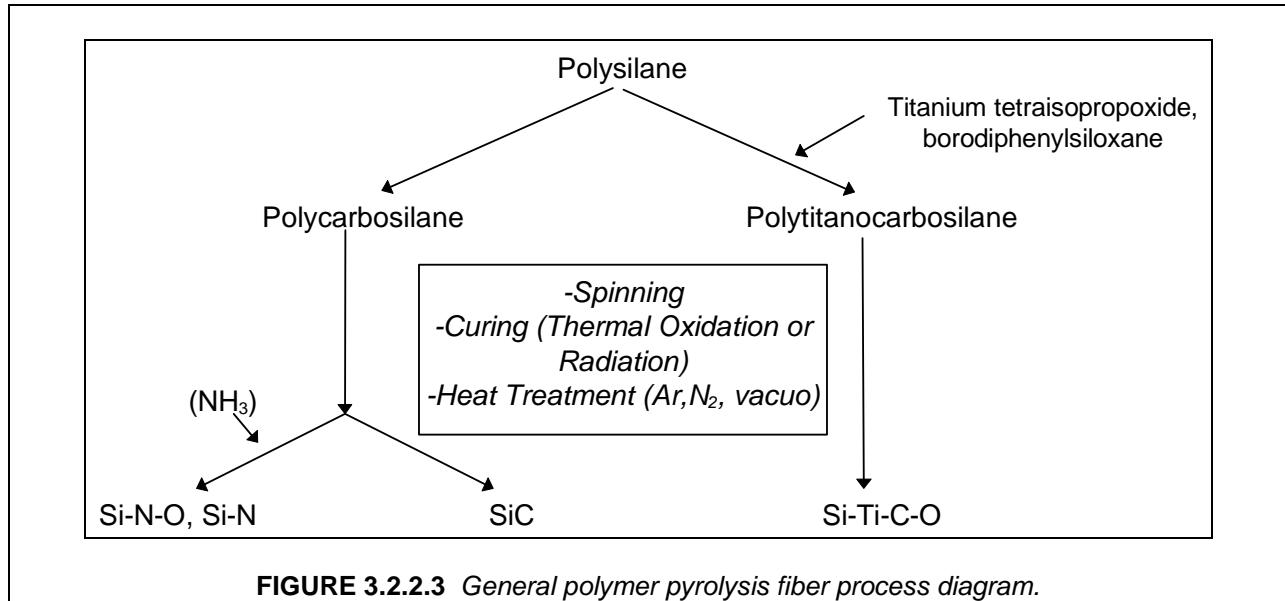
### 3.2.2.3 Small diameter SiC-based fibers

Today silicon-compound fibers that are commercially available in small diameters (less than 20 microns) and in multifilament form are generally synthesized by pyrolysis of organosilicon polymers. The manufacture of SiC-based fibers using the polymer pyrolysis (PP) process was first developed by Yajima, et al., by converting dimethyldichlorosilane (CH<sub>3</sub>)<sub>2</sub>SiCl<sub>2</sub>, to dodecamethylcyclohexasilane, [(CH<sub>3</sub>)<sub>2</sub>Si]<sub>6</sub>, which in turn is converted to an organosilicon polymer, (-Si-C-)<sub>n</sub> (References 3.2.2.3 (a) through (e)). The general process is shown in the flow diagram of Figure 3.2.2.3.

The viscous organosilicon liquid can be easily melt-spun into multifilament fibers of small diameter which are then subjected to a cure or cross-linking treatment in air or in ionizing radiation in order to produce a handleable "green" fiber. The green fibers are then converted to ceramic fibers by slowly heating them in inert or nitrogen containing atmosphere up to ~2192°F (~1200°C).

The final ceramic fibers obtained by the original polymer pyrolysis process had diameters in the range of 10-20 microns and were amorphous or microcrystalline with nanometer-sized grains of beta-SiC or Si<sub>3</sub>N<sub>4</sub> (Reference 3.2.2.3(e)). Their microstructures also contained varying amounts of free carbon, oxygen (probably combined as Si-O-C or Si-O-N), and microporosity. This resulted in fibers with densities and moduli significantly lower than those of the silicon compounds in pure, dense form. But more importantly, the fiber microstructures were thermally unstable above ~2192°F (~1200°C) due to decomposition

of the Si-C-O phase into SiO and CO gases (Reference 3.2.2.3(f)). As this decomposition occurs, large pores form within the fibers, resulting in a significant time/temperature-dependent loss in fiber strength. It has been demonstrated that this degradation can be delayed to higher temperatures by using approaches which do not allow the decomposition gases to escape or by significantly reducing the oxygen content within the as-processed fibers (References 3.2.2.3(g) and h)).



Several producers have modified the composition of their PP fibers for improved properties. Ube Industries provides "Tyranno" fibers in which approximately 2 wt% titanium is added to inhibit grain growth at elevated temperatures (Reference 3.2.2.3(i)). Tonen has developed fibers based on silicon nitride (Reference 3.2.2.3(j)), using silane, pyridine, and ammonia to produce a spinnable polymer. Dow Corning developed the HPZ fiber with an Si-N-O-C composition based on the pyrolysis of a hydridopolydisilazne polymer (Reference 3.2.2.3(k)).

Based on these developments in the late 80's and early 90's, producers of these silicon compound fibers have refined their chemistry and processing methods to produce silicon-compound fibers with lower oxygen content, improved Si-C stoichiometry and better control of grain size. These improvements in composition and microstructure have resulted in a new generation of silicon-compound fibers (Sylramic™, Hi-Nicalon, and Nicalon-S) with higher modulus and improved thermal stability.

Table 3.2.2.3 gives general information and representative properties for polymer pyrolysis fibers from different manufacturers.

### 3.2.2.4 Carbon fibers

#### 3.2.2.4.1 Introduction and applications

Carbon fibers are not new technology. They were produced by Thomas Edison as filaments for his electric light bulb. But in the 1960s carbon fibers were the first "high-strength, high modulus" fibers, developed for structural composites for the space and defense applications. Since the initial introduction of those fibers, the quality and performance have increased significantly and the price has gone down dramatically. As a result, carbon fibers are now used in a wide variety of commercial/industrial/consumer applications, such as sporting goods (golf clubs, tennis rackets, bicycle frames, arrows) composite hulls

for boats, aircraft brakes, automobile components, industrial rollers and storage tanks, furnace insulation, electronic component shielding, and windmill blades for wind energy systems.

**TABLE 3.2.2.3** *Properties for polymer pyrolysis fibers.*

Property/Fiber Name	Nicalon CG	Hi-Nicalon	Nicalon S	Sylramic*	Tyr-anno	HPZ	Tonen
Manufacturer	Nippon Carbon	Nippon Carbon	Nippon Carbon	Dow Corning	Ube	Dow Corning	Tonen
Fiber diameter	14	12	11	10	10	10	10
Density (g/cm <sup>-3</sup> )	2.55	2.74	3.10	3.0	2.50		
Tensile strength (GPa)	3.0	3.4	2.6	3.1	2.76		
Tensile modulus (GPa)	190	270	420	400	193		
Tensile elongation (%)	1.4	1.0	0.6	0.8	-		
Composition Si wt.%	56.6	62.4	68.9	69	SiTiCO	S-C-N	
C	31.7	37.1	30.9	29	-		
O	11.7	0.5	0.2	-	-		
C/Si (atomic)	1.31	1.39	1.05	1.0	-		
Crystallite size (nm)	<2	5 - 10	11	500	-		
Thermal stability (°C)	1110	1230	1450	1420	1300		

\* Additional experimental Sylramic fibers are available based on higher purity, well crystallized  $\beta$ -SiC for higher temperature, lower creep applications.

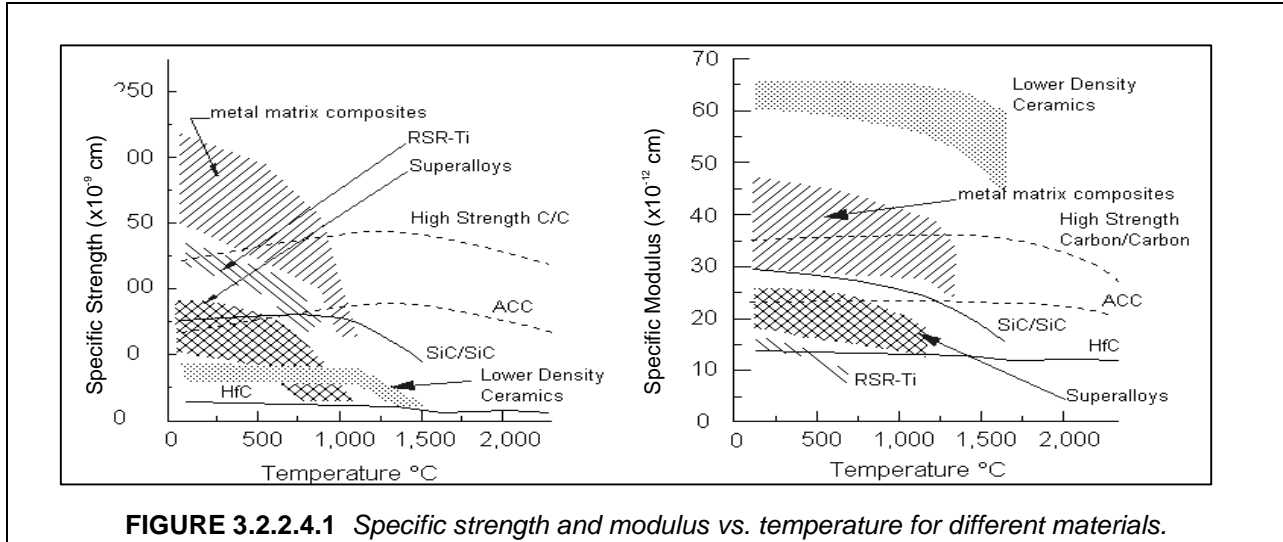
In all of these applications, carbon fibers offer high strength, high stiffness, low density, and low thermal expansion not achievable with any other structural fibers. In addition, carbon fibers have high thermal and electrical conductivity which make them suitable for specialized applications where thermal energy and electrical current need to be specifically controlled.

Carbon fibers have the unique characteristic of maintaining their mechanical properties up to temperatures (3992°F (2200°C)) where other materials (polymers, metals, and ceramics) have lost their mechanical integrity or have significantly softened. The one caveat on this high temperature stability is that it has to be under non-oxidizing conditions. Carbon fibers are subject to increasingly rapid oxidation at temperatures above 932°F (500°C). This oxidation susceptibility requires an oxygen-free environment, oxygen barrier protection, or a short duration performance requirements.

Carbon fibers are used in a variety of different composite matrix systems -- polymer, metals, carbon, and ceramic matrices. In each of these systems, the carbon fibers are the primary structural element, producing a composite with excellent strength and stiffness and reduced weight.

Carbon-carbon composites are a well-established technology used for high performance aircraft brakes, rocket exhaust nozzles, missile nose cones, and seals. The carbon-carbon composites have low densities and maintain their mechanical properties to significantly higher temperatures, as compared to other structural materials and illustrated in Figure 3.2.2.4.1.

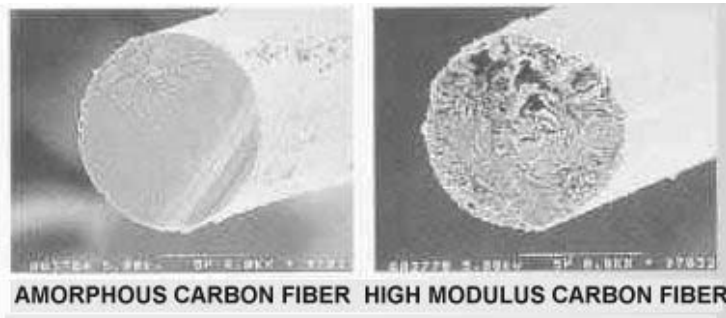




3.2.2.4.2 Structure and general properties of carbon fibers

Carbon fibers are commonly produced by the pyrolysis of hydrocarbon precursors in a non-reactive atmosphere. The initial conversion of the organic precursor to carbon in the pyrolysis process produces an amorphous crystal structure in the carbon fiber. High temperature heat treatment of the carbon fibers converts the amorphous structure to crystalline graphite. The purity, crystalline structure, grain size, and defect structure are key factors in determining the properties of carbon fibers. The extent and orientation of the carbon crystal structure are tailored to provide the desired mechanical, thermal and electronic properties. Highly crystalline atomic structures provide the highest modulus, highest thermal conductivity, and lowest electrical resistivity.

Carbon fiber tensile strength is primarily controlled by the defect level, and modulus is controlled by crystalline orientation and degree of crystallinity. Thus, by varying those process parameters that can influence orientation and crystallinity (e.g., precursor purity, stretch ratios, overall process tension levels, heat treatment furnace temperature,) in the manufacture of both the precursor and the carbon fiber, the resulting carbon fibers can have a broad a broad range of properties (Table 3.2.2.4.2). Generally, carbon fibers are classified into three major grades -- standard or aerospace grade, intermediate modulus, and high modulus.



Generally, carbon fibers are classified into three major grades -- standard or aerospace grade, intermediate modulus, and high modulus.

To date, most sales and qualification of carbon fibers for aerospace applications have been with the standard-grade carbon fiber, and use of this type of fiber is likely to continue as long as the present applications continue in production. The newer aerospace (and some recreation) applications, however, are primarily involved with the intermediate modulus, and high modulus fibers.

Carbon fibers possess the highest specific modulus (modulus/density) of all commercially available reinforcing fibers (several times that of conventional metals). However, since commercial PAN-based carbon fibers were first introduced 20 years ago, their physical properties have improved dramatically. In fact, over the past decade improvements in precursor as well as carbon fiber technology have increased the tensile strength of this class of fibers from approximately 500 ksi (3.45 GPa) to 1000 ksi (6.9 GPa). Today, specific strengths (tensile strength/density) achievable with PAN-based carbon fibers are among

the highest of all commercially available reinforcing fibers. They also have a very good balance of properties.

**TABLE 3.2.2.4.2** Major grades of carbon fibers.

Grade	Modulus (Msi / GPa)	% Strain to Failure	Thermal Conductivity (W/mK)	Electrical Resistivity (micro-ohm-m)
Standard / Aerospace	~33 / 230	~2.0	~8	18
	~37 / 255	~2.0	~14	11
Intermediate Modulus	~60 / 414	~1.5	~120	8.5
High Modulus	>100 / 758	<1.0	~520	2.5

A wide variety of fibers are now commercially available that range in tensile modulus from 30 Msi (207 GPa) to 120 Msi (800 GPa) with elongation from less than 1 percent to over 2 percent. Finally, a substantial amount of progress has been made in the ability to convert carbon fibers into intermediate product forms. The fibers can be braided, woven, knitted, and converted into unidirectional tapes that ultimately can be processed into composites. These materials, however, are inherently more difficult to handle than conventional textile fibers and conventional processing speeds are not practical with them. Very specialized techniques are therefore continually being developed to improve processing efficiencies.

Pitch fibers are used for applications requiring high modulus and high thermal conductivity and, perhaps, increased resistance to oxidation. On the other hand, the small crystallite size typical of PAN-based carbon fibers provides higher strength for intermediate-modulus, high-strength applications.

One of the unique features of carbon fibers is that the coefficient of thermal expansion along the axis is effectively zero or slightly negative. This is an effect of the graphite crystal structure which is oriented along the axis. The degree of crystallization and orientation directly determines the axial thermal expansion. Carbon fibers with a high degree of crystallinity and alignment have negative axial thermal expansions with values as great as  $-1.5$  ppm/ $^{\circ}$ C. The negative axial thermal expansion of the carbon fibers is used in composites to produce low expansion components for use in mirrors and precision alignment devices. Refer to Reference 3.2.2.4.2 for additional information on the structural and general properties of carbon fibers.

#### 3.2.2.4.3 Fabrication of carbon fibers

Commercial carbon fibers are produced by a two step process -- the spinning of an organic precursor and a thermal conversion to carbon. There are three types of precursors used on a commercial basis to manufacture carbon fibers: rayon, polyacrylonitrile (PAN), and pitch. Rayon, the raw material used for the first high-performance carbon fibers, was the dominant precursor material in the 1960s and early 1970s. However, because of its low carbon yield (2~30 percent), high processing cost, and limited physical properties, today rayon precursor is used for less than 1 percent of commercial carbon fibers (Reference 3.2.2.4.2).

#### PAN-based Carbon Fibers

PAN-based carbon fibers are derived from polyacrylonitrile or acrylic copolymers. Normally, a solution of the polymer is either wet or dry spun into PAN precursor fibers that are ultimately converted into carbon fibers; Figure 3.2.2.4.3(a) gives a flow diagram for the process. The precursor fibers used by the major PAN-based carbon fiber suppliers differ significantly from the acrylic fibers used for textile acrylic

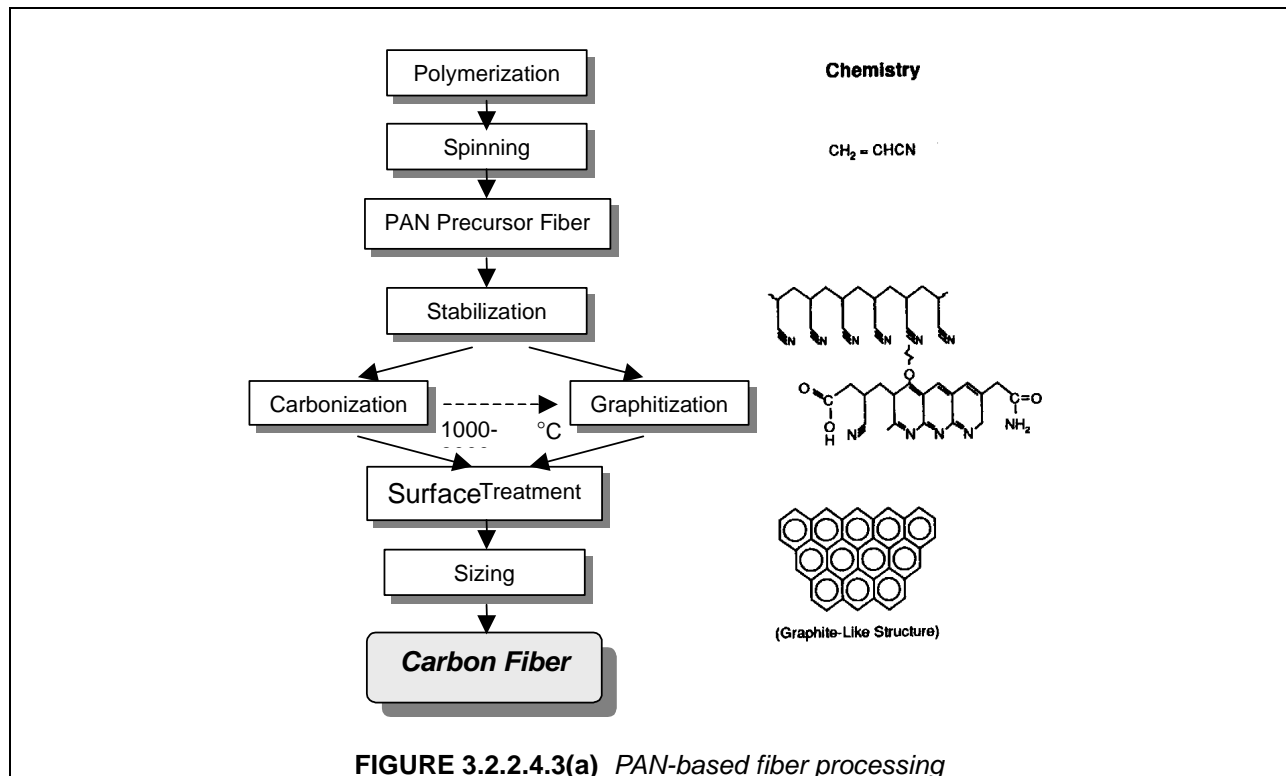
apparel and industrial applications. PAN precursor fibers have fewer filaments per tow stage, a higher level of purity, smaller filament diameter, and higher acrylonitrile (AN) content (i.e., normally greater than 90 percent AN) than fibers used in textile applications. Also, the polymer composition and molecular weight must be modified to produce the desired carbon fiber properties. Because of these differences, the cost of acrylic precursor fibers is three to five times greater than that of acrylic fibers used in textile applications.

Commercial production of PAN-based precursor fibers is based on solution spinning of PAN polymer. Typically, a dilute solution of acrylic polymer is extruded into a coagulation bath (wet spinning) or a hot-gas environment (dry spinning). The use of large amounts of solvents is a fundamental factor in the production process, which results in environmental as well as product design limitations.

Stabilization of PAN involves heating the fiber in air to temperatures ranging from 392 to 572°F (200 to 300°C) for approximately 1 hour. The stabilization treatment is followed by carbonization in an inert atmosphere at temperatures greater than 2192°F (1200°C). Orientation of the graphite-like crystal structure, and thus the fiber modulus, can be further increased by heat treatment (termed graphitization) at temperatures up to 5450°F (3000°C). The continuous carbon or graphite fiber is then surface treated and coated with a sizing agent prior to winding the continuous filaments on bobbins. The surface treatment is an oxidation of the fiber surface to promote adhesion to the matrix resin in the composite, and the size promotes handleability and wettability of the fiber with the matrix resin.

#### Pitch-based carbon fibers (Reference 3.2.2.4.2)

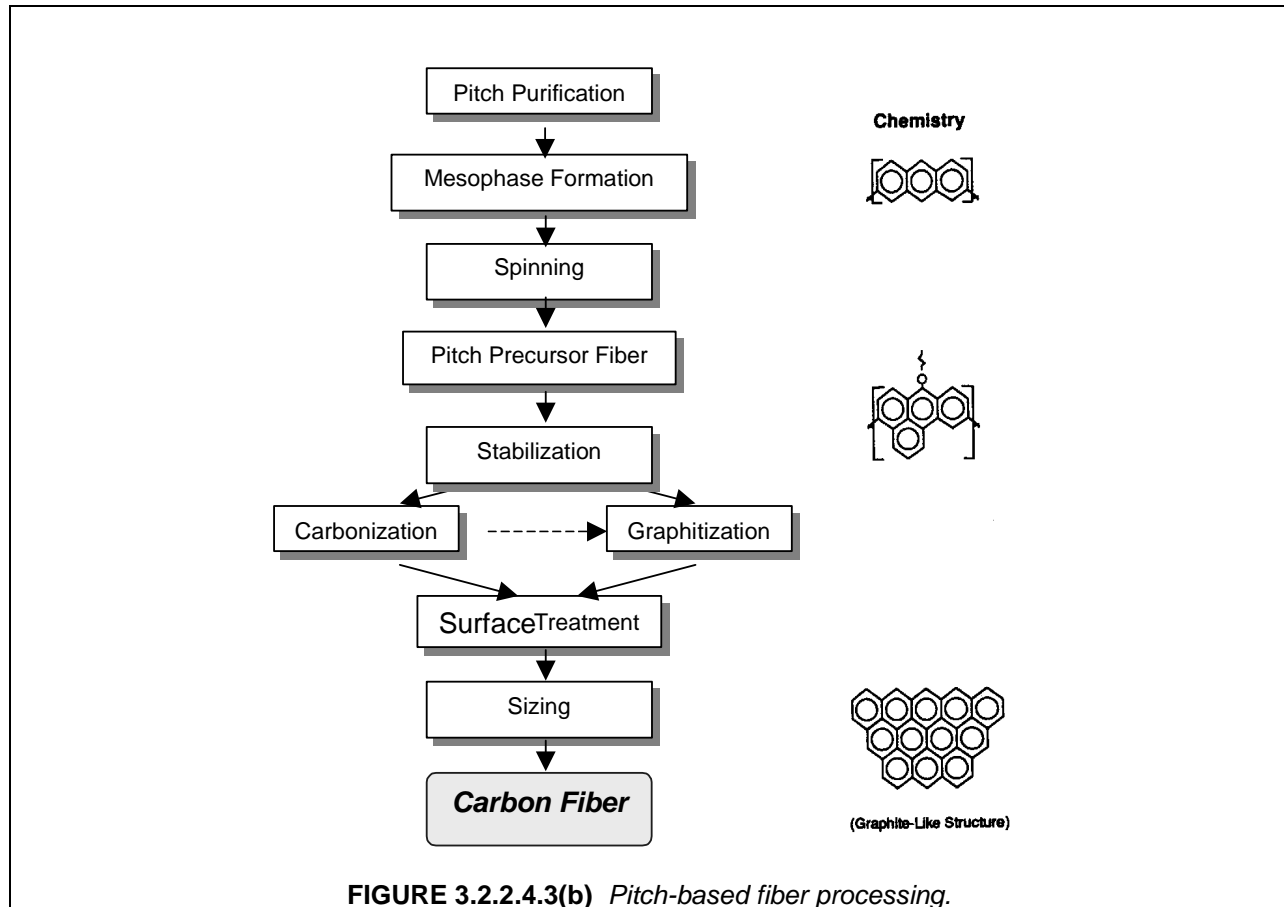
The production of high-modulus, pitch-based carbon fibers begins by heat treating a petroleum or coal tar pitch feedstock to produce a liquid crystal precursor, termed "mesophase." The liquid crystal material is melt spun into a precursor fiber that is converted into a carbon fiber in a process similar to that used for PAN-based carbon fiber. The high degree of molecular orientation of the as-spun fiber allows it, unlike PAN, to develop a truly graphitic crystalline structure during the carbonization/ graphitization step. The fundamentals of the process are shown in Figure 3.2.2.4.3(b).



**FIGURE 3.2.2.4.3(a)** PAN-based fiber processing

Potential advantages for pitch-based carbon fibers include economics and unique properties that result from their high degree of molecular orientation. Since the starting carbon content for pitch precursor is significantly higher than PAN precursor (approximately 93 percent versus 68 percent), the theoretical yield for pitch fibers is substantially higher than that of PAN fibers. Also, acrylonitrile, the raw material for PAN-based carbon fibers, costs almost twice as much as the raw pitch used to produce pitch-based carbon fibers. These differences should make pitch-based fibers much less expensive than PAN-based carbon fibers. However, today's selling prices for these two varieties of carbon fibers are equivalent for comparable performance fibers. However, in the future it is probable that at least some portion of this potential cost advantage will be realized.

Pitch-based carbon fibers exhibit the highest specific modulus (tensile modulus/density) among the commercially available reinforcing fibers. Presently, pitch-based carbon fibers are available with moduli as high as 140 Msi (965 GPa). Their graphite structure also allows pitch-based fibers to possess excellent axial conductivities (both electrical and thermal) and very low axial coefficient of thermal expansion. Commercial pitch-based fibers are available with thermal conductivities that are three to four times that of copper. Because of this, pitch-based carbon fibers are preferred for space applications where stiffness, thermal expansion, or conductivity are critical performance requirements. However, the extended graphite structure of present commercial pitch-based fibers also makes them more sensitive to fiber surface defects and structural flaws. This increased sensitivity causes the tensile strength of these fibers to be 40 to 50 percent lower than that of PAN-based carbon fibers. Also, it results in a compressive strength for pitch-based carbon fibers that is only one-third that of PAN-based fibers.



In the area of pitch-based carbon fibers, the technical focus is directed toward higher tensile strength products, improved compressive properties, improved routes to mesophase production, higher conductivity, and improved processability. It should be noted that only pitch-based carbon fibers (and vapor-grown carbon fibers) have the graphite crystal structure needed to develop high thermal conductivity; this property creates a unique market for this fiber. Improved mesophase production techniques are a key to both improved process economics as well as improved products. The other key is reducing the flaw sensitivity of the fiber. Recent research shows that by modifying the microstructure of the pitch-based fiber during the spinning process, without significantly altering the crystalline orientation, it is possible to improve both the fiber tensile strength (reduced flaw sensitivity) and the compressive strength. Even though commercial pitch-based fibers have reached 93 percent of the modulus of perfect graphite, their tensile strength is less than 4 percent of that predicted by theory. Obviously, considerable improvement in the tensile strength of pitch-based carbon fibers is possible. Additional information on pitch-based carbon fibers is available from Reference 3.2.2.4.2.

#### 3.2.2.4.4 Availability and sources of carbon fibers

Carbon fibers are produced across a range of diameters from 5-15 microns. Multifilament tows/rovings have filament counts ranging from 1,000 to 48,000 with linear densities ranging from 0.066 to 3.20 grams/meter. Standard spools contain from 1000 to 5000 meters of tow. Tows are commonly provided with sizing at the 1-1.5 wt% level to provide for handling and weavability. Woven fabrics are commercially available from weaving companies and composite supply companies in a range of different weaves (plain, satin, twill, basket, etc.). The major producers of carbon fiber are shown in Table 3.2.2.4.4.



**TABLE 3.2.2.4.4** Major producers of carbon fiber.

Producer	U.S. Location	WEB Address	Phone
BP Amoco	Alpharetta, GA	<a href="http://www.bpamococarbonfibers.com">http://www.bpamococarbonfibers.com</a>	800-222-2448
Conoco	Houston, TX	<a href="http://carbonfiber.conoco.com/index_dynamic.html">http://carbonfiber.conoco.com/index_dynamic.html</a>	877-319-8853
Grafil	Irvine, CA	<a href="http://www.grafil.com">http://www.grafil.com</a>	800-365-5533
Hexcel	Salt Lake City, UT	<a href="http://www.hexcelfibers.com">http://www.hexcelfibers.com</a>	800-987-0658
Nippon Graphite	Cypress, CA (T. Wong)	<a href="http://plaza6.mbn.or.jp/~NGF/english/ngf.htm">http://plaza6.mbn.or.jp/~NGF/english/ngf.htm</a>	714-826-3235
Toho Carbon	Menlo Park, CA	<a href="http://www.tohorayon.co.jp/rayon/eng/index_4.html">http://www.tohorayon.co.jp/rayon/eng/index_4.html</a>	650-614-0765
Toray Carbon	Santa Ana, California	<a href="http://www.toray.com/cfa/fiber.htm">http://www.toray.com/cfa/fiber.htm</a>	714-431-2320
Zoltek	St. Louis, MO	<a href="http://www.zoltek.com">http://www.zoltek.com</a>	314- 291-5110

#### 3.2.2.4.5 Specific properties of carbon fibers

The following tables lists property data for different carbon/graphite fibers. Data are taken from product brochures and producer literature (September 2000)

**TABLE 3.2.2.4.5** *Properties of carbon specific fibers, continued on next page (References 3.2.2.4.5(a) through 3.2.2.4.5(c)).*

Source / Type	Strength		Modulus		Tensile Strain %	Thermal Cond. (W/m•K)
	ksi	GPa	Msi	GPa		
<b>BP AMOCO</b>						
T-300	545	3.75	33	231	1.4 %	8
T-650	620	4.28	37	255	1.7 %	14
P-25	200	1.38	23	159	0.9%	22
P-30X	300	2.07	30	207	0.7%	50
P-55S	275	1.90	55	379	0.5%	120
P-75S	300	2.10	75	517	0.4%	185
P-100S	350	2.41	110	758	0.3%	520
P-120S	350	2.41	120	827	0.3%	640
<b>CONOCO</b>						
<b>GRAFIL</b>						
34-700	650	4.50	34	234	1.9%	
34-600	600	4.15	34	234	1.8%	
TR-30	640	4.41	34	235	1.9%	
TR-40	680	4.70	34	235	2.0%	
TR-50	710	4.90	35	240	2.0%	
TR-H50	710	4.90	37	255	1.9%	
MR35E	640	4.41	43	295	1.5%	
MR40	640	4.41	43	295	1.5%	
MR50	790	5.40	42	290	1.8%	
MS40	670	4.61	50	345	1.3%	
HR40	640	4.41	57	390	1.1%	
HS40	640	4.41	66	450	1.0%	

**TABLE 3.2.2.4.5** *Properties of carbon specific fibers, continued on next page (References 3.2.2.4.5(a) through 3.2.2.4.5(c)).*

Source / Type	Strength		Modulus		Tensile Strain %	Thermal Cond. (W/m•K)
	ksi	GPa	Msi	GPa		
<b>HEXCEL</b>						
AS4C	610	4.20	33.5	231	1.8%	
AS4D	660	4.55	35	241	1.9%	
IM4	650	4.48	40	276	1.6%	
IM4A	650	4.48	42	290	1.5%	
AS4	602	4.15	33	228	1.8%	
IM6	799	5.51	40.5	279	2.0%	
IM7	737	5.08	40	276	1.4%	
IM7 (5000 Spec)	802	5.53	40	276	2.0%	
IM7 (6000 Spec)	835	5.76	42	292	2.0%	
IM8	809	5.58	44	304	1.8%	
IM9	887	6.12	44	304	4.8%	
UHM	518	3.57	64	440	0.8%	
<b>NIPPON - GRANOC</b>						
XN-05	156	1.13	8	55		4.7
XN-10	230	1.66	17	115		-
XN-15	303	2.19	23	160		6.3
XN-20	378	2.73	29	200		13
XN-35	502	3.63	50	350		-
XN-50	484	3.50	72	500		-
XN-50A	560	3.83	75	520	0.7	-
XN-60	484	3.50	87	600		-
XN-70	484	3.50	101	700		-
XN-70A	530	3.63	105	720	0.5	-
XN-80	484	3.50	115	800		320
XN-80A	530	3.63	114	785	0.5	-
XN-85A	530	3.63	120	830	0.4	-
XN-90	470	3.40	128	890		500
YS-50A	560	3.83	75	520	0.7	-
YSH-50A	530	3.83	75	520		120
YSH-60A	530	3.83	91	630		180
YSH-70A	502	3.63	104	720		250
YS-80A	502	3.63	113	785		320
YS-90A	488	3.53	127	880		500
YS-95A	488	3.53	133	920		600
HT grade	664	4.80	33	230		9.4
HM grade	609	4.40	54	377		69

**TABLE 3.2.2.4.5** *Properties of carbon specific fibers, concluded (References 3.2.2.4.5(a) through 3.2.2.4.5(c)).*

Source / Type	Strength		Modulus		Tensile Strain %	Thermal Cond. (W/m•K)
	ksi	GPa	Msi	GPa		
<b>TOHO</b>						
HTA	515	3.72	34	235	1.7 %	
UT500	666	4.81	35	240	2.0 %	
IM400	624	4.51	43	295	1.5 %	
IM600	801	5.79	41	285	2.0 %	
LM16	435	3.14	22	155	2.0 %	
HM35	407	2.94	50	345	0.9 %	
UM40	678	4.90	55	380	1.2 %	
UM46	650	4.70	63	435	1.1 %	
UM55	529	3.82	78	540	0.7 %	
UM63	488	3.53	88	610	0.6 %	
UM68	461	3.33	94	650	0.5 %	
<b>TORAY</b>						
T300	512	3.53	33.4	230	1.5 %	
T300J	611	4.21	33.4	230	1.8 %	
T400H	640	4.41	36.3	250	1.8 %	
T800S	600	4.14	33.4	230	1.8 %	
T700S	711	4.90	33.4	230	2.1 %	
T700G	711	4.9	34.8	240	2.0 %	
T800H	798	5.49	42.7	294	1.9 %	
T1000G	924	6.37	42.7	294	2.2 %	
M35J	683	4.70	49.8	343	1.4 %	
M40J	640	4.41	54.7	377	1.2 %	
M46J	611	4.21	63.3	436	1.0 %	
M50J	697	4.12	69.0	475	0.8 %	
M55J	583	4.02	78.2	540	0.8 %	
M60J	569	3.92	85.3	588	0.7 %	
M30S	796	5.49	42.7	294	1.9 %	
M30G	739	5.10	42.7	294	1.7 %	
M40	398	2.74	56.9	392	0.7 %	
<b>ZOLTEK</b>						
Panex 30	500	3.60	30	207	1.5%	
Panex 33	550	3.80	33	228	1.6%	

### 3.2.3 High temperature properties of continuous ceramic fibers

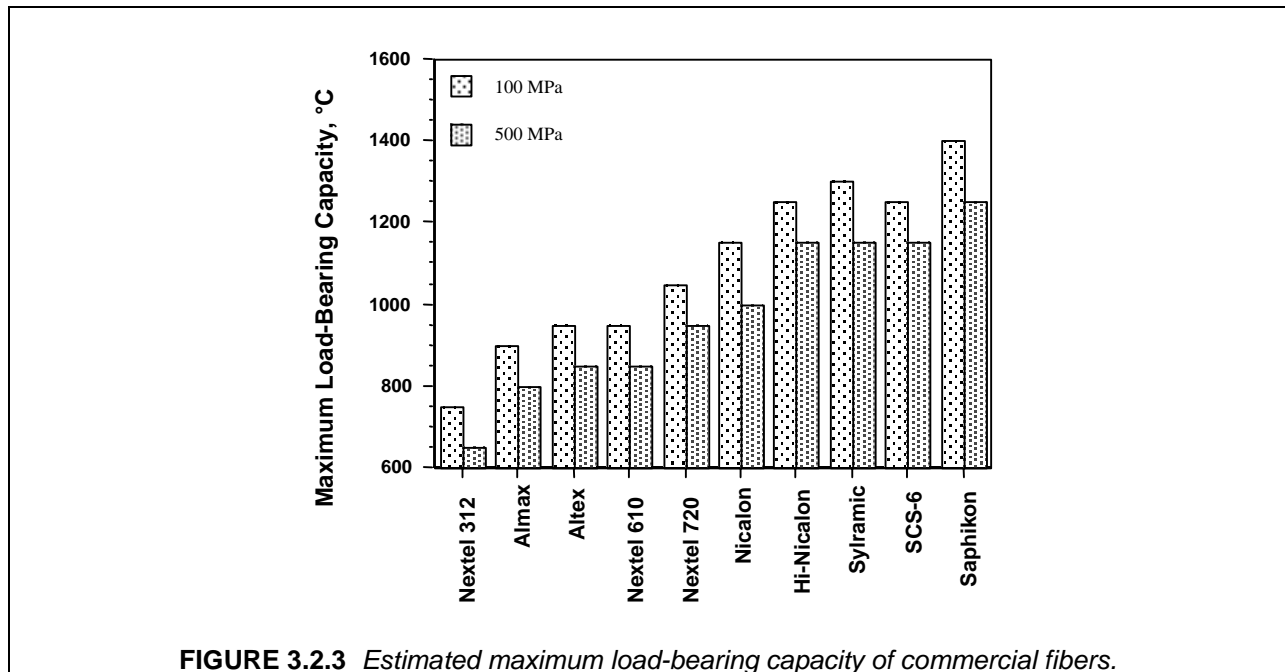
The properties of ceramic fibers at high temperature determine their suitability for their use as reinforcements in CFCCs. Even under zero stress conditions, most commercial fibers, oxide and non-oxide,



experience strength loss for short time exposures of  $\sim 2192^{\circ}\text{F}$  ( $\sim 1200^{\circ}\text{C}$ ), whether during composite fabrication or use. Strength degradation is related to a number of factors, including grain growth, either of existing phases or during the crystallization of new phases, thermally-activated growth of flaws, or decomposition of non-equilibrium phases in the fiber. For polycrystalline  $\text{Al}_2\text{O}_3$ -based fibers, strength degradation appears to be primarily related to grain growth and defects associated with this grain growth. For the small diameter Si-based fibers produced by polymer pyrolysis, Si-C-O phases within the fiber begin to decompose, creating flaws and eventually porosity.

The resistance to deformation and time-dependent rupture are critical in the selection of continuous ceramic fibers for use in CMCs. Many CFCC use conditions involve structural loading of the fibers over long time periods which can range from 100 hours to more than 10,000 hours. Even in “non-structural” applications, fibers can be subjected to high thermal stresses caused by thermal gradients within the composite. It is generally accepted that only small stress-induced dimensional changes can be tolerated, typically less than 1% over its service life. A second criterion is stress rupture life, which in many cases can be substantially less than time to a 1% strain limit. Rupture life can be dominated by a number of factors relating to fiber composition and microstructure, including slow crack growth from existing flaws or creep cavitation.

Figure 3.2.3 gives estimates for the maximum load-bearing temperature of commercial fibers in CFCCs. These estimates were determined by DiCarlo using measurements of creep rate and creep rupture life on single filaments using the assumption that maximum fiber use temperature is limited either by fiber rupture or by excessive creep strain (assumed to be 1%). To illustrate the effect of service stress, maximum temperatures are shown both for a fiber stress of 100 MPa (14 ksi), which is assumed to be the minimum experienced in a CFCC and for 500 MPa (72 ksi), which is representative of structurally loaded components. Of course, the maximum use temperatures for fibers are dependent on a variety of assumptions. Adverse environmental effects (e.g., the presence of alkali) may reduce temperature capacity substantially; in contrast, in composites where the matrix contributes to load-bearing capacity (e.g. CVD SiC), temperature capacity may increase. The maximum load-bearing temperature is dependent on stress; temperature capability is reduced by  $\sim 212^{\circ}\text{F}$  ( $\sim 100^{\circ}\text{C}$ ) for both SiC and  $\text{Al}_2\text{O}_3$ -based fibers as stress is increased from 100 MPa to 500 MPa.



It is clear that, as a group, polycrystalline  $\text{Al}_2\text{O}_3$ -based fibers have much lower maximum load-bearing temperature than the SiC-based fibers. None of these fibers can withstand 500 MPa above 1000°C for times greater than 100 hours. Among the  $\text{Al}_2\text{O}_3$ -based fibers, temperature capability is improved by eliminating  $\text{B}_2\text{O}_3$  additions (e.g., from Nextel 312 to Altex), and improved further by additions of creep-resistant phases such as mullite (e.g., from Nextel 610 to Nextel 720). The highest performance oxide fiber is the Saphikon single crystal fiber, which has no measurable creep below 2912°F (1600°C), allowing it to outperform even the SiC-based fibers.

The maximum load-bearing temperature of SiC-based fibers is 2192-2372°F (1200-1300°C). Hi-Nicalon, Sylramic, and SCS-6 are all in this range, which is surprising given their very different microstructure, chemical compositions and complicated mechanism controlling deformation. Load bearing capacity is limited by the 1% creep limit in each case. The Nicalon fiber has lesser load bearing capacity due to the low viscosity of its oxygen-containing amorphous second phase.

Fiber manufacturers are examining new and improved processing approaches which attempt to eliminate or minimize microstructural sources for strength degradation and high temperature creep. For example, SiC fibers are being produced which demonstrate improved strength retention and creep performance relative to commercially-available fibers. This has been accomplished through achieving stoichiometric or nearly stoichiometric SiC microstructures, the elimination of second phases such as Si-C-O that enhance creep and creep-related flaw growth, and increasing grain size. For oxide fibers, improvements in creep have been demonstrated by oxide compositions more creep-resistant than alumina, such as mullite and YAG. Grain growth inhibitors will probably also be required for oxide fibers to keep grain sizes small and to pin grain boundary motion for additional improvement in fiber creep resistance.

#### 3.2.3.1 Carbon fibers

Reserved for future use.

#### 3.2.4 Discontinuous reinforcements – whiskers, particulates, and in-situ

Reserved for future use.

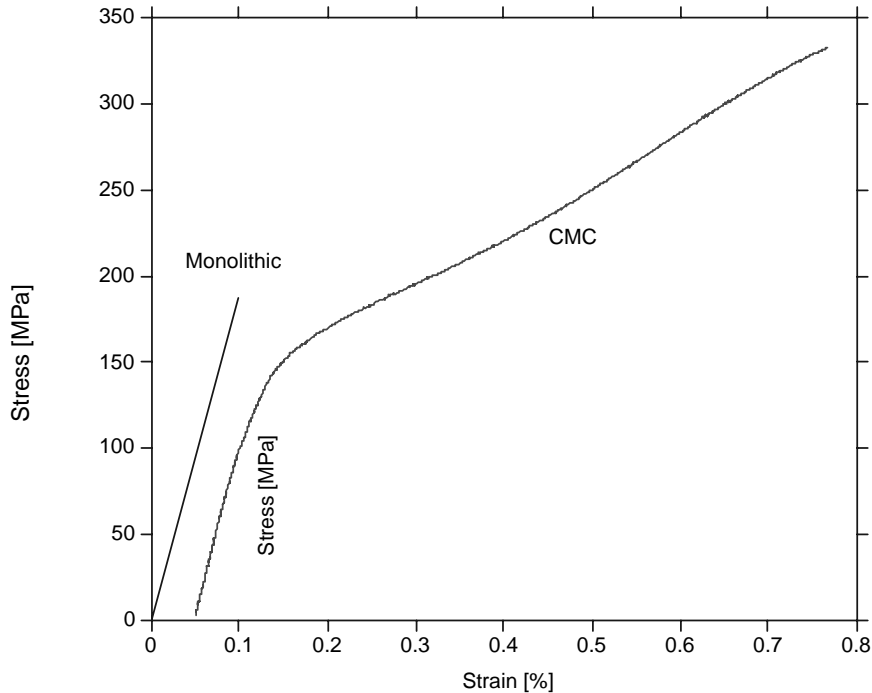
### 3.3 INTERPHASE/INTERFACE TECHNOLOGY AND APPROACHES

#### 3.3.1 Introduction

The fiber/matrix interface plays a significant role in determining the fracture behavior and mechanical properties of ceramic matrix composites. The critical driving force in the development of fiber reinforced ceramic composites has been the benefit of graceful failure and damage tolerance. In contrast to brittle ceramics, ceramic composites can survive local damage and inelastic deformation without catastrophic failure. In simpler terms, they have greater strain capability than monolithic ceramics, as shown in Figure 3.3.1(a), where the stress-strain curve has a nonlinear response at higher strain levels.

By combining the two materials in the fiber reinforced composite, the strength of the composite is stronger than the strength of the matrix by itself. And the composite has higher strain capability than either of its constituents by themselves.

By introducing a high strength, high modulus fiber into a low strength, low modulus matrix, the material designer increases the strength of the composite, because load is transferred to the high strength fibers. The effectiveness of the load transfer depends on the difference in moduli between the fiber and the matrix, the volume fraction and architecture/distribution of the fibers, the length of the fibers, and the interfacial bond between the fibers and the matrix (Reference 3.3.1(a)).



**FIGURE 3.3.1(a)** *Illustrates the individual mechanical response of a monolithic ceramic compared with a high strength fiber reinforced composite with a lower strength matrix.*

But the fiber reinforced ceramic composites also have a higher strain capability than either of the constituents. From a practical perspective, the composites have “pseudo-ductility” similar to the ductile response of common metals. Nonlinear stress-strain in metals is a function of plastic deformation, commonly produced by dislocation mechanisms. In contrast, fiber reinforced ceramic composites develop nonlinear mechanical response based on three underlying mechanisms, as described by Zok, et al., (Reference 3.3.1(b)). The three mechanisms are frictional dissipation at the fiber-matrix interface, matrix cracking to increase the elastic compliance, and stress redistribution produced by matrix cracking. Implicit in these mechanisms is the assumption that the cracks in the matrix are bridged to some degree by the fibers.

From a material design perspective (as described by Faber (Reference 3.3.1(c)) and by Kerans, et al., (Reference 3.3.1(d)), there are two key issues in fiber reinforced ceramic composites -- how to deflect matrix cracks around the fiber and how to control the sliding resistance of the fiber in the matrix. Historically, the direct introduction of a ceramic fiber into a ceramic matrix has commonly produced a brittle composite, because there is a strong chemical bond between the fiber and the matrix. With a strong chemical bond, matrix cracks penetrate directly into the fiber with no crack deflection.

The predominant approach in developing the desired weak bond at the fiber-matrix interface has been to use a thin (0.1-5 micron) coating around the ceramic fibers to develop the desired crack deflection and the required sliding friction. The coating acts as a debond layer and as a “low-friction” sliding

interface. This sounds simple, but the functionality of interface coatings actually depends on many complex factors.

- Chemical bonding at the interfaces with the matrix and with the fiber.
- Inherent toughness of the interface relative to the fiber toughness.
- Surface roughness of the substrate fibers.
- Residual stresses produced during fabrication based on differential thermal expansion coefficients.
- Shear planes and shear strength of the interface.

### 3.3.1.1 *The roles and requirements for fiber interfaces and coatings*

The primary role of the fiber interface coating in CMCs is to provide a mechanism for crack deflection through debonding and frictional sliding. A successful fiber coating will stop or deflect microcracks at the fiber matrix interface. Without debonding, the matrix cracks will cut through the ceramic fibers, rather than deflecting around the high-strength reinforcements. As further stress is applied and microcracks begin combining and opening, the weakly bound fibers bridge the cracks and slide in the matrix, dissipating strain energy. Without controlled sliding of the fiber in the matrix, strain energy will not be dissipated and high stresses will form as the crack advances, leading to fiber fracture. For more detailed information, please refer to References 3.3.1.1(a) - (c).

The interface coatings should be chemically and microstructurally stable at the desired operational conditions. Ideally, the coating will have no significant changes in chemical composition, phase content or grain size at high temperatures or with repeated temperature cycling. (The exception are coatings in which microcracking or phase change is desired to control the mechanical response to stress).

The coating should be thermodynamically stable with the fiber substrate and the surrounding matrix, resisting solid-state reactions. Lastly, the coating should have resistance to oxidation, corrosion, steam and reduction attack. Given the critical role that the interface plays in strength and fracture toughness, any significant susceptibility of the coating to environmental attack across the range of performance conditions is a critical application barrier.

Interface coatings also serve secondary functions in CMCs. Composite fabrication conditions and matrix precursors may be chemically aggressive with high temperatures and reactive chemical species, such as halides and free radicals. Interface coatings can act as reaction and diffusion barriers for the underlying ceramic fibers, preventing chemical attack and fiber degradation. In a similar manner, multilayer coating systems may require top level barrier layers to prevent attack on susceptible coating layers. To that end, barrier coatings should be thermodynamically stable, crack and pore free, and be deposited in a uniform and controllable manner.

Ceramic fibers in CMCs may also be susceptible to environmental attack and solid-state matrix interactions in high temperature performance conditions. Fiber interface coatings can serve as reaction and diffusion barriers in aggressive operations, again protecting the underlying fibers and/or intermediate coatings from chemical attack.

Coating deposition and heat-treatment conditions may, in their own right, be chemically and/or thermodynamically aggressive. Any interface coating developed or deposited on a ceramic fiber should be compatible with the fiber microstructure, composition, and physical properties.

Residual and/or thermal stresses in ceramic interface coatings can have negative effects on crack deflection. Under the ideal conditions, the thermal expansion of interface coatings will be equivalent or close to the fiber thermal expansion. That match will minimize stresses developed during fabrication or in operation.

### 3.3.1.2 Fabrication of fiber interface coatings

The fabrication of thin (0.5 micron) coatings on ceramic fibers is a significant fabrication challenge. Coatings deposition techniques must be controllable and tailorable, producing fiber coatings that are uniform in composition, thickness and morphology. This is particularly challenging for multifilament tows and 2-D and 3-D weaves, where the fine diameter (<20 microns) fibers are tightly bundled and in direct contact. It is critically important that all the ceramic fibers are uniformly coated, because uncoated fibers can fracture at low stresses and are susceptible to oxidation.

Coating deposition processes should be highly reproducible between and within lots, providing uniform coatings across and with multifilament tow, 2-D fabric, and 3-D weave and braid geometries. Ideally, the coating process should be continuous in nature, able to deposit coatings on long lengths of tows and woven fabrics.

Interface fiber coatings for ceramic composites have been produced through two approaches -- in-situ chemistry and direct application coatings. For in-situ fabrication, chemical species react at the fiber surface to form the desired composition or morphology. (An example of in-situ chemistry is the formation of carbon rich layers on silicon oxycarbide fibers in glass-ceramic composites). The chemical species which form can be present in the composite by its inherent composition or by deliberate additives to the fiber or the matrix. In all cases, the formation of the desired interface requires very precise heat-treatment, depends on diffusion mechanisms and uniform distribution of the reactants, and is not easily controlled for reproducibility.

Direct application of the desired coating to the fiber before composite fabrication is the preferred coating method. The composition, morphology, and thickness of the coating can be easily controlled and tailored, independent of later composite fabrication conditions and heat treatments. The primary direct coating methods for ceramic fibers are chemical vapor deposition and liquid precursors (sol-gel and polymer precursors) coatings. Both of these methods are advantageous for coating multifilament geometries, with good penetration into the interior of filament bundles. Additional techniques have been investigated -- line-of-sight techniques, electrochemistry, and electroless plating.

Chemical vapor deposition (CVD) uses vapor transport of gaseous species which react locally and deposit the reaction product on the fiber substrate. The deposition reaction is driven by thermal, radio-frequency (RF), plasma, or photon energy sources. Control of the reaction chemistry and the resulting coating composition and uniformity depends on careful management of the reactant chemistry, partial pressures of reactants, gas flow rates, temperature uniformity, and local diffusion conditions within the fiber architecture.

As described in the section of CVD composite processing, a wide range of ceramic compositions can be deposited by CVD -- carbides, nitrides, and oxides. Coating thicknesses can range from nanometers to several micrometers, depending on deposition rates and times.

Liquid precursor coating techniques depend on immersion and coating of the fine fibers with an aqueous or organic based liquid which contains the chemical species of interest. Evaporation of a solvent or chemical reaction of the liquid species leaves behind a thin coating on the fibers which is subsequently reacted or heat-treated to produce the desired ceramic coating.

Sol-gel coatings use metal alkoxides in alcohol-water solvent mixtures. Hydrolysis of the alkoxides produces a metal-oxide-metal gel which is dried to remove the solvent and the reaction products. The dried gel is thermally converted to a dense oxide. Silicon, aluminum, zirconium, and titanium alkoxides are commonly available, and are suitable for producing single and mixed oxide ceramic compositions. The major benefit of sol-gel coating is that the coating and conversion process is relatively low temperature and benign. The primary limitation of sol-gel coatings is the high shrinkage volume which occurs during drying and conversion, limiting the coating thickness that can be produced without shrinkage cracks. Thicker coatings can be developed by multiple coating steps.

Pre-ceramic polymers commonly consist of carbon and silicon based oligomers with reactive groups which can be cross linked by thermal, catalytic, or energetic means. The pre-ceramic polymer is coated on the fiber in either neat or dilute solutions, followed by polymer curing. The cured polymer coating is then heat-treated in a controlled atmosphere to produce the desired ceramic composition. Carbon, silicon carbide, silicon nitride, and oxycarbides have been produced by this method. Nitride compositions are formed by the incorporation of imides and amide groups in the polymer or by pyrolysis of the cured polymer in ammonia atmospheres. Additional metallic elements can be incorporated, based on the availability of precursor polymers with the desired elements, such as aluminum, zirconium, titanium, etc. Polymer precursor coating is particularly suitable for carbide and nitride coatings, compared to sol-gel which is more suitable for oxide coatings. Polymer coatings are also limited in coating thickness, because of the shrinkage that occurs during the pyrolysis step. As with sol-gel coatings, thickness can be built up with multiple coatings.

Line-of-sight techniques refer to methods based on sputtering, physical vapor deposition, ion implantation, and electron-beam evaporation. All these techniques are dependent on a straight direct path from the material source to the target substrate. With that limitation, they can be used for large diameter (>40 micrometers) ceramic fibers, but have very limited use for multifilament tows and weaves, where line-of-sight into the tows or weaves is intermittently blocked.

Deposition of metallic coatings on ceramic fibers can be done by electrolytic and electroless deposition techniques. In both methods, metals are deposited from solutions. In electrolysis, metal ions are reduced and deposited on a cathode substrate by the application of a voltage across an anode and a cathode. The ceramic fibers can be electrically conductive by their composition (silicon carbide or carbon) or by a flash metal deposition technique. Electrolytic metal coatings can be deposited across a full range of metals -- copper, nickel, chromium, zinc, tin, lead, silver, gold, platinum etc. However, most of these metals do not have high temperature oxidation stability, are very dense, or are very high cost.

In electroless coating, metal ions are deposited from solution through the action of a chemical agent in a redox reaction without passage of an electrical current. They involve reduction of a complexed metal using a mild reducing agent. Electroless coatings can be deposited in nickel, copper, silver, gold, and platinum.

### **3.3.2 Interphase composition**

#### *3.3.2.1 Carbon*

The initial success with ceramic matrix composites in the 1970's and 80's was based on the use of carbon and boron nitride as interface coatings. Graphitic carbon interface coatings are deposited on fiber tows, cloth, or preforms using chemical vapor deposition/infiltration at relatively low temperatures of approximately 1832°F (1000°C). Different precursor gases have been used to produce the graphitic carbon layers; propylene-, ethylene-, and methane-derived graphitic carbon coatings have been produced at similar temperatures. Differences in deposition rate and coating texture are achieved for the same processing temperatures. Propylene-derived graphitic coatings will typically have a highly aligned graphitic structure (with the (002) axis preferentially aligned perpendicular to the fiber surface), whereas ethylene- and methane-derived coatings tend to be polycrystalline with randomly oriented, small grains. Amorphous and/or graphitic carbon interlayers can also be applied to fibers using different resin-based techniques.

Ceramic composites with carbon interfaces have excellent room temperature mechanical properties. It has been hypothesized that since graphite possesses an extremely low modulus along the c-axis, the interface is compliant and lessens the interfacial stresses that occur due to thermal expansion differences between the fiber and the matrix. The interfacial shear strength is dependent on the thickness of the carbon layer; thicker coatings produce lower interfacial shear strengths. The optimum thickness for the carbon interfaces in most composites is 0.25-0.30 microns. Carbon works well for most low temperature, non-corrosive CFCC applications. However, carbon begins to oxidize at temperatures as low as 797°F (425°C), and oxidation is rapid at temperatures greater than 1562°F (850°C). When a carbon-containing

ceramic matrix composite is exposed to an oxidizing environment, oxidation begins with the attack/loss of carbon interfaces at exposed fiber ends. Carbon is removed along the entire fiber length (the rate of carbon loss will depend on the temperature) resulting in an open channel at the fiber-matrix interface. The surfaces within the channel (fiber and matrix) will subsequently oxidize to form silica (at least in the SiC/SiC system) which eventually bonds the components together. Now, the interfaces are "filled" with a brittle, fully bonded material which does not permit debonding and fiber sliding, resulting in brittle fracture. Oxidation of the fiber surfaces will also degrade the fiber properties leading to significant losses in composite strength.

### 3.3.2.2 *Boron nitride*

The poor oxidation resistance of carbon has led to the examination of alternative fiber coatings for use in CFCCs. Hexagonal boron nitride possesses a crystallographic structure and mechanical properties similar to those of graphitic carbon. Most important is the significant improvement in oxidation resistance attained by using BN coatings instead of carbon. Pyrolytic BN coatings exhibit good oxidation resistance up to temperatures as high as 2192°F (1200°C). However, the presence of water vapor significantly accelerates the decomposition of BN over a wide temperature range. BN can be deposited on fiber tows, cloth, or preforms via chemical vapor deposition and infiltration techniques, usually from either BCl<sub>3</sub> or BF<sub>3</sub>, NH<sub>3</sub>, and hydrogen. Different BN processing temperatures can yield different crystallographic structures in the coating; deposition temperatures less than 1652°F (900°C) result in amorphous BN, intermediate temperatures produce fine-grained, polycrystalline, hexagonal BN, and high deposition temperatures, greater than 2372°F (1300°C), result in highly aligned, large-grain sized BN. The deposition temperature used will also depend on the fiber reinforcement selected. The BN crystalline structure has a profound affect on the stability of the BN coating in corrosive environments; amorphous coatings are inherently unstable and will decompose even at relatively low temperatures, whereas the highly crystalline BN deposited at high temperatures is much more stable in environments containing oxygen and water vapor at elevated temperatures. The purity of the BN is also an important factor; oxygen incorporated in the BN lattice during processing can be detrimental to the long term stability since it can cause reactions with non-oxide fibers and matrices. High purity BN has greater stability at higher temperatures. BN can be intentionally doped with various elements to increase the stability. Recent experiments with Si-doped BN have shown improvements in the oxidation resistance of these composites. High-temperature, high purity BN shows the most promise for use in CFCCs in corrosive environments.

### 3.3.2.3 *Oxide*

Oxide fiber coatings offer an advantage of thermodynamic stability in the most frequent use environments anticipated for CFCCs - high temperature and oxidizing. It should be noted from the start that oxide fiber coatings are generally applied to oxide fibers and nonoxide coatings to nonoxide fibers. The development of oxide fiber coatings has lagged that of nonoxide coatings such as boron nitride for a number of reasons: (1) nonoxide fibers have been superior to oxide fibers and coating development has generally advanced with improvements in fibers, (2) promising layered oxide compositions are not chemically compatible with available oxide fibers, (3) there has been difficulty identifying nonlayered oxide fiber compositions which are stable with and weakly bonded to the available oxide fibers, and (4) the greater chemical complexity of the oxide compositions, compared to nonoxide coatings such as carbon and boron nitride, has made coating deposition more difficult.

#### 3.3.2.3.1 *Non-layered oxide interfaces*

There has been recent progress in identifying nonlayered oxides which appear chemically stable with available oxide fiber compositions, such as alumina and mullite, and which exhibit weak bonding to the fibers. These oxide fiber coatings would generally be easier to incorporate into a composite than the layered oxides because no preferential alignment of the grains within the coating would be necessary to achieve weak interfaces. The initial compounds which were identified as exhibiting weak bonding to alumina include the rare-earth phosphates of the general formula Me<sub>3</sub>+PO<sub>4</sub>. This includes the monazite family of minerals comprised of the larger rare-earth elements of the lanthanide series (La, Ce, Pr, Nd, Pm,

Sm, Eu, Gd, and Tb). It also includes the xenotime family of minerals comprised of scandium, yttrium and the smaller rare-earth elements of the lanthanide series (Dy, Ho, Er, Tm, Yb, and Lu). Additional compounds of the general formula  $ABO_4$  formula are being investigated as well. These include the broad family of tungstate ( $Me_2+WO_4$ ) and tantalates ( $Re_3+TaO_4$ ). Composites fabricated with monazite ( $LaPO_4$ ) and scheelite ( $CaWO_4$ ) fiber coatings have shown good debonding from Nextel 610 and Nextel 720 fibers.

These  $ABO_4$  compounds are the newest interface concepts for oxide-oxide CFCCs, and as a result, are just beginning to receive detailed study and to be incorporated into real world composites. It appears at this time that careful control of the sol-gel precursor chemistry used for depositing these coatings is required to prevent fiber strength degradation. Substantial research efforts to verify the effectiveness of this interface concept at elevated temperature for extended periods of time are still required.

### 3.3.2.3.2 Layered oxide interfaces

Layered oxide fiber coatings have received a high level of interest because it is hoped that they could mimic the easy debonding observed with the layered nonoxides carbon and boron nitride which have work well in non-oxide CFCCs (although only under nonoxidizing use environments for long times). Early work focused on sheet silicates known as fluormicas such as  $KMg_{2.5}(Si_4O_{10})F_2$  and  $KMg_3(AlSi_3O_{10})F_2$ . These compounds created easy delamination along interfaces, however, their complex chemistry did not allow for a chemically stable interface and work on these compounds was discontinued. Simpler chemistry layered oxides such as beta-aluminas ( $Me_1+Al_{11}O_{17}$ ) and magnetoplumbites ( $Me_2+Al_{12}O_{19}$ ) are now being investigated. Hibonite,  $CaAl_{12}O_{19}$ , has been shown to promote interfacial debonding in model composite systems with sapphire monofilaments. Use of this interfacial approach in real composites has been limited for a couple of reasons: (1) temperatures greater than the capability of commercially available fibers have been required to form and texture the layered structures (although there has been recent progress at lowering the temperatures) and (2) the layered compounds are not chemically stable with the highest temperature capability fiber, Nextel 720. Higher temperature capability, nonsilicate fibers, will likely be required in order to utilize layered oxides as fiber coatings in real world composites.

### 3.3.2.3.3 Porous matrices and porous/fugitive coatings

Because of the difficulty in identifying promising oxide fiber compositions, there has also been a significant effort at developing CFCCs which do not rely on fiber coatings, but instead rely on significant matrix porosity to create a weakly bonded fiber-matrix interface or rely on a gap at the fiber-matrix interface created by a fugitive interface coating. This section summarizes the various interphase approaches being pursued for oxide-oxide CFCCs.

#### 3.3.2.3.3.1 Porous matrices

Many organizations have developed a damage tolerant oxide-oxide CFCC by avoiding fiber coatings altogether. These composite systems have matrices with a large amount of residual porosity, often times exceeding 40 percent. These matrices are generally very weak and as a result, the composites usually exhibit damage tolerance through progressive and distributed damage under off-axis loading. Axial loading of these composites generally produce fairly linear stress-strain curves since their properties are fiber dominated. The ultimate strains-to-failure are representative of that expected from unreinforced fiber tows. These porous matrix CFCCs are generally the furthest developed and characterized oxide-oxide systems because of their relative ease of fabrication as a result of not requiring a separate fiber coating step. Composites of this type are being fabricated by General Electric, Composite Optics Inc., Boeing-McDonnell Douglas and 3M. Table 3.3.2.3.3.1 summarizes the properties of these systems.

These composite systems offer low costs and attractive axial properties and damage tolerance. Because of the low matrix strength, the composite systems all exhibit low interlaminar strengths. Because there is no fiber-matrix interphase, a potential deficiency in this type of composite systems is the possibility of reaction or sintering between matrix particles and the fibers at points where contact is made between the two leading to fiber strength degradation. These composite systems may be limited in the time



and/or temperature of their exposure in use in order to prevent a reduction in the composite properties. Lack of a fiber coating with a porous matrix may also subject the fibers to corrosive species within the use environment causing fiber strength degradation.

**TABLE 3.3.2.3.3.1** *Properties of 0°/90° Nextel fibers.*

<b>Oxide Based Fibers</b>	Nextel 312	Nextel 610	Nextel 650	Nextel 720
Property at room temperature				
Nominal Composition	Alumino-borosilicate	Alumina	Alumina w/ Zirconia	Alumina-Mullite
Density (g/cm <sup>3</sup> )	2.70	3.88	4.10	3.40
Tensile Strength (ksi)	24.6	42.5	36.9	30.4
Young's Modulus (Msi)	21.7	54.1	51.9	37.7
Maximum Use Temperature (°F) = 80% strength retention	--	1472	2012	2282
Thermal Conductivity [Btu in/s-ft <sup>2</sup> °F]	≈ 5.0x10 <sup>-3</sup> (est)	≈ 5.7x10 <sup>-2</sup> (est)	NA	≈ 1.9x10 <sup>-2</sup> (est)
Coef. of Linear Thermal Expansion (ppm/K)	3.0 212-1832°F	7.9 212-1832°F	8.0 212-1832°F	6.0 212-1832°F
Specific Heat [J/(g-k)]	≈ 0.7	≈0.9	NA	≈ 0.85

Information supplied by 3M September 2000

### 3.3.2.3.3.2 *Porous Coatings*

A logical extension of the porous matrix concept is to localize the porosity in a thin layer close to the fiber. Researchers have evaluated porous oxides (ZrO<sub>2</sub>, Y<sub>2</sub>O<sub>3</sub>, and Al<sub>2</sub>O<sub>3</sub>) as coatings on sapphire fibers in an alumina matrix. The porosity (at the 30 vol.% level) in the coatings produced the desired debonding, but there was high sliding stress and limited fiber pull-out. It is suspected that the crack path in the porous interface was too rough and convoluted, producing the high sliding stresses. It was proposed that tailoring of the grain size and porosity in the interface could reduce the sliding stress to acceptable levels.

### 3.3.2.3.3.3 *Fugitive Interfaces*

The fugitive interface coating concept relies on the processing of the composite with a carbon coating in place, which after composite densification can be oxidized away to create a void along the fiber-matrix interface. In essence, the interface is debonded from the start rather than relying on the stress intensity at the front of a propagating crack to initiate the interfacial debonding. This interface approach has received substantial investigation because of the ease and low cost of depositing carbon coatings by both sol-gel and CVD processes. Substantial development work has been performed at the Air Force Wright Laboratory which showed that some load transfer still occurs between the matrix and the fibers even though the interface is debonded. The amount of load transfer will depend on the thickness of the carbon coating relative to the roughness of the fiber and the variation in the diameter along the fiber length. The coating concept also yields composite systems which will likely be limited in their time and temperature of use since fiber strength degradation may occur over time at points where fibers and matrices are in contact (they must be at places where load transfer occurs). Sintering of the matrix over time will also result in additional points of contact between fibers and matrix. These possible fiber degradation mechanisms could be minimized through careful selection of fiber-matrix combinations. This interface concept also

leaves the fibers exposed to the use atmosphere which could result in fiber degradation under harsh use environments.

These different approaches to interfaces offer a wide range of options in developing an effective coating approach. Although the basic concepts for an effective interface are obvious, there remain significant challenges in both the fundamental and practical levels. From a functional perspective, a compliant, debonding interface coating/layer with moderate sliding stress and high temperature oxidation resistance is the performance goal. However, the coating will have little practical application unless it can be fabricated on tows and weaves (2-D and 3-D) in a controllable, repeatable, low cost method. The goal of the interface community is to develop a coating system that has the following properties:

- Controllable bonding to the desired fiber and the designated matrix.
- Tailored mechanical and thermal expansion properties to produce crack deflection and controlled sliding stress.
- Chemical and mechanical stability in the composite under high temperature (1200°C) oxidizing/corrosive operational conditions.
- Low compliance to compensate for residual and misfit stresses in the composite.
- Controlled, repeatable, low cost deposition on tow and weave with no fiber bridging with minimal environmental impact.

### 3.3.3 Other

Reserved for future use.

## 3.4 FABRICATION AND FORMING OF FIBER ARCHITECTURES

### 3.4.1 General

The aerospace and related industries have a critical need for composite materials that are stronger, lighter, more durable, corrosion resistant, and perform at elevated temperatures (Reference 3.4.1). Ceramic matrix composites (CMCs) are meeting these requirements through selective design of the fiber, matrix, and interface control mechanism. The design problem being addressed will define the process, material system(s), and application specific requirements for the end product. This section addresses a generalized overview of the fiber forms, how they are used, and where they are commercially available.

### 3.4.2 Fiber architectures

Fiber material forms include low crimp uniweave, 2-D and 3-D integrally woven shapes, and 2-D/3-D braided preforms (References 3.4.2(a) and (b)). Each of the general classes above have their own distinct advantages and disadvantages and are briefly described in Table 3.4.2 (Reference 3.4.2(a)). An illustration showing various weave architectures is shown in Figure 3.4.2. The proper use and selection of fibrous preforms should effect the end application in one or more of the following areas: lower cost, reduce joints, reduce fasteners, simplify part design, increase design flexibility, improve impact, and damage tolerance.

The design flexibility and number of available fiber systems have led to an increased application of preforms in CMCs. In general, a fiber with a reasonable degree of flexibility and surface lubricity can be processed. However, as the modulus and fiber diameters increase, some limitations on weaving speeds and/or fiber radii of a z-directional reinforcement will exist narrowing the fabrication window.

#### 3.4.2.1 Uniweaves

The low crimp uniweaves are a highly unbalanced (~98% warp, ~2% fill) preform used to approximate the properties of a uni-tape with some of the handling advantages of the fabric. Low crimp uniweaves are

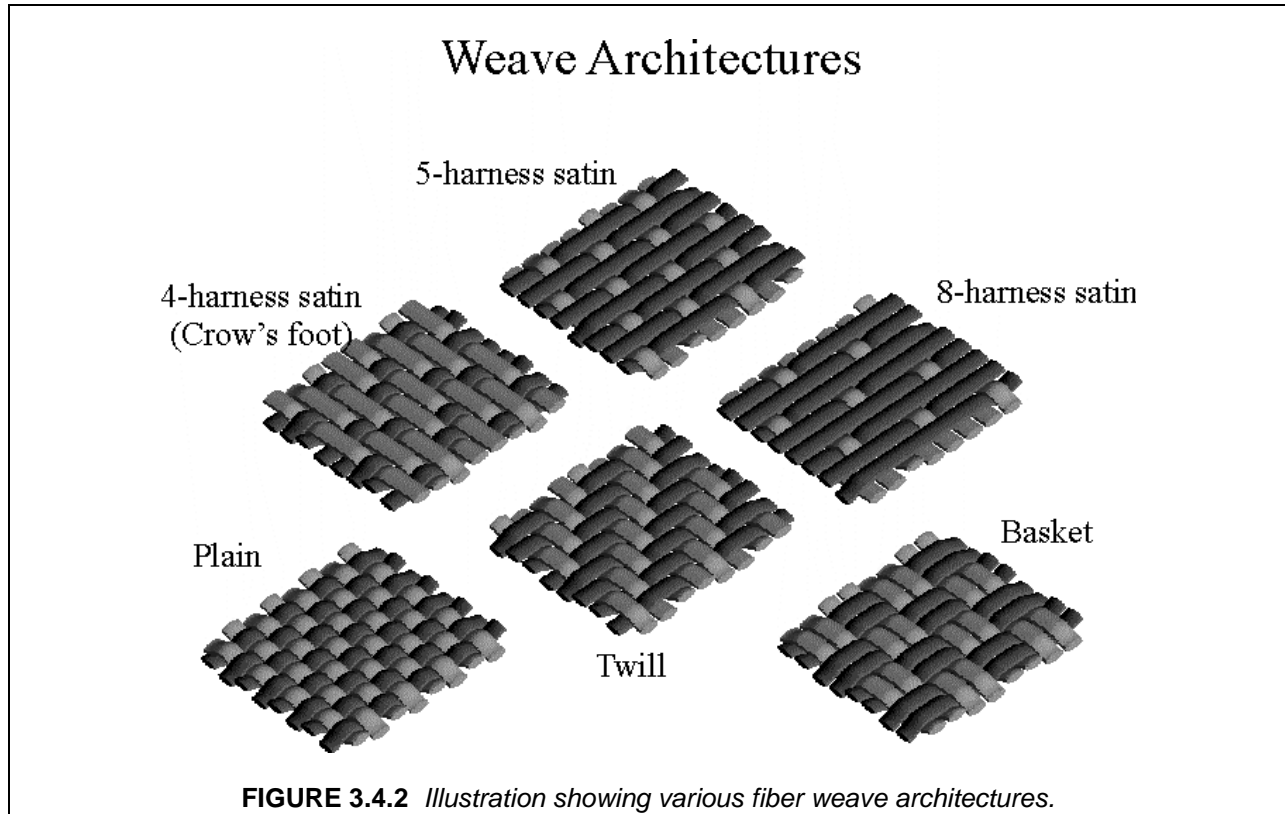
used where high in-plane properties are required. The uniweave will also allow significant usage of high modulus fibers in the warp direction.

**TABLE 3.4.2** *Preform advantages and disadvantages.*

<b>Preform Type</b>	<b>Advantages</b>	<b>Disadvantages</b>
Uniweave	<ul style="list-style-type: none"> <li>• High In-Plane Properties</li> <li>• Good Design Flexibility</li> <li>• Automated Process</li> </ul>	<ul style="list-style-type: none"> <li>• Low Transverse and Out-of-Plane Properties</li> <li>• Poor Fabric Stability</li> </ul>
2-D Woven Fabric	<ul style="list-style-type: none"> <li>• High In-Plane Properties</li> <li>• Excellent Batch-to-Batch Consistency</li> <li>• Often Most Economical Choice in Balance of Properties</li> <li>• Good Drapability</li> <li>• Highly Automated Process</li> <li>• High Design Flexibility for Complex Shapes</li> </ul>	<ul style="list-style-type: none"> <li>• Low Transverse and Out-of-Plane Properties</li> </ul>
3-D Woven Fabric	<ul style="list-style-type: none"> <li>• Moderate In-Plane and Out-of-Plane Properties</li> <li>• Good Drapability</li> <li>• Automated Process</li> </ul>	<ul style="list-style-type: none"> <li>• Limited Off-Axis Properties</li> </ul>
2-D Braided Fabric	<ul style="list-style-type: none"> <li>• Good Balance in Off Axis Properties</li> <li>• Good Drapability</li> <li>• Complex Parts</li> <li>• Automated Preform Process</li> </ul>	<ul style="list-style-type: none"> <li>• Low Out-of-Plane Properties</li> <li>• Size Limitations</li> <li>• Tend to be Limited to Axisymmetric Shapes</li> <li>• High set-up costs</li> </ul>
3-D Braided Fabric	<ul style="list-style-type: none"> <li>• Good In-Plane and Out-of-Plane Properties</li> <li>• Good Drapability</li> <li>• Highly Automated Process</li> </ul>	<ul style="list-style-type: none"> <li>• Slow Preform Process</li> <li>• Size Limitations</li> <li>• Tend to be Limited to Axisymmetric Shapes</li> <li>• High Set-Up Costs</li> <li>• Difficult Reproducibility, i.e., Fiber Content</li> </ul>

### 3.4.2.2 2-D woven architectures

The 2-D woven fabrics are more commonly used where good in-plane properties, drapability, large area coverage are required. 2-D fabrics are commonly used in fabrication of engine nozzle structure, thermal protection systems, and other relatively non-complex hardware with relatively low out-of-plane loading requirements.



### 3.4.2.3 3-D architectures

There are basically three types of 3-D preforms currently used and they are polar, orthogonal, and angle interlock. The three basic fiber directions can be customized forming a continuous structure tailored to specific design requirements. The 3-D structure has excellent damage tolerance, improved shear property performance, and designed specific strength and stiffness.

#### 3.4.2.3.1 3-D polar architectures

Polar preforms include axial, radial, and circumferential fiber orientations. These weaves are used for applications having a body of revolution (cylinders, throat components, nose tip, exit cones, convergent and divergent shapes). Non axisymmetric shapes (leading edges, conic/rectangular transitions) can be made by forming and then slitting the preform to the desired dimensions.

#### 3.4.2.3.2 3-D orthogonal architectures

3-D orthogonal preforms are defined by the 90 degrees intersection of all the fibers to one another. Parts made from this process are typically formed in a billet and then subsequently machined to final dimension. These preforms are typically used in relative flat applications (brakes, rotors, and heat sinks).

#### 3.4.2.3.3 3-D angle interlock architectures

The 3-D angle interlock preforms are used where part complexity can be reduced by integrally woven parts such as seals, heat exchangers, stiffeners, truss structures, and related composite inserts (such as tee clip and pi clip). An application typically allows for some reduction in in-plane properties while improving the out-of-plane properties. These fiber preforms are commonly used in conjunction with the 2-D

weaves. The near-net shapes commonly reduce fabrication costs normally associated with laying up multiple plies of a 2-D fabric.

#### 3.4.2.4 2-D braided architectures

The 2-D braided preforms are highly automated and a good balance of off-axis properties can be achieved with the 2-D triaxial configuration ( $\theta+f$ ). 2-D braided preforms are well suited for complex applications (aircraft ducting and tubing, aircraft engine containment, propeller blades, missile nose cones, and satellite components.)

#### 3.4.2.5 3-D braided architectures

The unique feature of the 3-D braided preform is the ability to provide through the thickness reinforcements. The process lends itself to solid and/or hollow inserts and attachment cross sections of varying geometries (Reference 3.4.2(b)).

### 3.4.3 Fabric weave and braid manufacturers

A list of ceramic fiber preform manufacturers (2D and 3D weavers and braiders), including locations and telephone numbers, is given in Table 3.4.3.

**TABLE 3.4.3** Weave and braid manufacturers (March 2000).

Preform Manufacturer	Location	Telephone Number
Albany International Research Co.	Mansfield, MA	(508) 339-7300
Albany International Techniweave Co	Rochester, NH	(603) 335-2115
Atlantic Research Corp.	Gainesville, VA	(703) 754-5000
Bally Ribbon Mills	Bally, PA	(610) 845-2211
Fabric Development, Inc.	Quakertown, PA	(215) 536-1420
Fiber Concepts, Inc.	Conshohocken, PA	(610) 834-3234
Fiber Innovations, Inc.	Walpole, MA	(508) 660-2622
Fiber Materials, Inc.	Biddeford, ME	(207) 282-5911
Hexcel Corporation	Austin, TX	(512) 326-2995
Intec Products, Inc.	Anaheim, CA	(714) 630-9192
Lydall Manning	Ft. Washington, PA	(215) 542-2433
3M Ceramic Fiber Products	St. Paul, MN	(612) 736-3627
Pull-Rope Company, Inc.	Colorado Springs, CO	(719) 391-1892
Textile Products, Inc.	Anaheim, CA	(714) 761-0401
Textron Systems	Wilmington, MA	(978) 657-2961

### **3.5 EXTERNAL PROTECTIVE COATINGS**

Reserved for future use.

#### **3.5.1 External coating functions**

Reserved for future use.

##### *3.5.1.1 Environmental protection*

Reserved for future use.

##### *3.5.1.2 Thermal management*

Reserved for future use.

##### *3.5.1.3 Wear and abrasion*

Reserved for future use.

##### *3.5.1.4 Signature control*

Reserved for future use.

##### *3.5.1.5 Aerodynamic surface control*

Reserved for future use.

#### **3.5.2 Compositions and method of fabrication**

Reserved for future use.

##### *3.5.2.1 Compositions and structure*

Reserved for future use.

###### *3.5.2.1.1 Oxide compositions*

Reserved for future use.

###### *3.5.2.1.2 Non-oxide compositions*

Reserved for future use.

###### *3.5.2.1.3 Multilayer coatings*

Reserved for future use.

###### *3.5.2.1.4 Particulate composite coatings*

Reserved for future use.

##### *3.5.2.2 Methods of fabrication*

Reserved for future use.

#### *3.5.2.2.1 Chemical vapor deposition*

Reserved for future use.

#### *3.5.2.2.2 Thermal spray*

Reserved for future use.

#### *3.5.2.2.3 Physical vapor deposition*

Reserved for future use.

#### *3.5.2.2.4 Sinter/glaze coatings*

Reserved for future use.

#### *3.5.2.2.5 Diffusion and reaction sintering*

Reserved for future use.

### **3.5.3 Engineering considerations**

Reserved for future use.

#### *3.5.3.1 Thermodynamic compatibility*

Reserved for future use.

#### *3.5.3.2 Coating process compatibility*

Reserved for future use.

#### *3.5.3.3 Mechanical compatibility*

Reserved for future use.

##### *3.5.3.3.1 Thermal expansion match*

Reserved for future use.

##### *3.5.3.3.2 Coating strength*

Reserved for future use.

##### *3.5.3.3.3 Coating adhesion*

Reserved for future use.

##### *3.5.3.3.4 Strain accommodation*

Reserved for future use.

##### *3.5.3.3.5 Residual stresses*

Reserved for future use.

*3.5.3.4 Component geometry coatability*

Reserved for future use.

*3.5.3.5 Environmental stability of coating*

Reserved for future use.

**3.5.4 Examples of external coatings for CMCs**

Reserved for future use.

**3.6 CHARACTERIZATION METHODS (CHEMICAL AND MICROSTRUCTURAL)**

Reserved for future use.

**3.6.1 Bulk composite**

Reserved for future use.

*3.6.1.1 Composition*

Reserved for future use.

*3.6.1.2 Density*

Reserved for future use.

*3.6.1.3 Porosity*

Reserved for future use.

*3.6.1.4 Microstructure*

Reserved for future use.

*3.6.1.5 Defects*

Reserved for future use.

*3.6.1.6 Other physical*

Reserved for future use.

**3.6.2 Fibers/reinforcement**

Reserved for future use.

*3.6.2.1 Composition*

Reserved for future use.

*3.6.2.2 Density*

Reserved for future use.



3.6.2.3 *Porosity*

Reserved for future use.

3.6.2.4 *Microstructure*

Reserved for future use.

3.6.2.5 *Defects*

Reserved for future use.

3.6.2.6 *Sizing*

Reserved for future use.

3.6.2.7 *Slipping*

Reserved for future use.

3.6.2.8 *Other physical*

Reserved for future use.

**3.6.3 Matrices**

Reserved for future use.

3.6.3.1 *Composition*

Reserved for future use.

3.6.3.2 *Density*

Reserved for future use.

3.6.3.3 *Porosity*

Reserved for future use.

3.6.3.4 *Microstructure*

Reserved for future use.

3.6.3.5 *Defects*

Reserved for future use.

3.6.3.6 *Other physical*

Reserved for future use.

### 3.6.4 Interfaces

Reserved for future use.

#### 3.6.4.1 Composition/chemical phase

Reserved for future use.

## 3.7 NONDESTRUCTIVE EVALUATION METHODS FOR CMC (DEFECT CHARACTERIZATION)

A variety of non-destructive testing (NDT) techniques are available for detecting both surface and interior flaws in composites. Visual inspection and liquid penetrant methods can be used for identifying surface defects, while more sophisticated techniques are required for detecting internal flaws (i.e. voids, delamination, foreign inclusions, disbonds). These techniques include ultrasonics, radiography, thermography, acoustic emission, X-ray and eddy-current testing. The basic principles and procedures for these methods are covered in the MIL-HDBK-728 series, while more specific information on the theory and interpretation of data can be found in the following:

MIL-HDBK-731 Thermography  
 MIL-HDBK-732 Acoustic Emission  
 MIL-HDBK-733 Radiography  
 MIL-HDBK-787 Ultrasonic

These documents do not discuss the recent advances in NDT techniques, which is currently an active field of research and development.

Nondestructive Evaluation (NDE), Nondestructive Testing (NDT) and Nondestructive Characterization (NDC), while having similar meanings, have subtle but different applied results. NDT is a long-standing subset of NDE and has usually been considered to be the application of one of several well defined NDT methods: X-ray radiography (usually referred to as Radiographic Testing, RGT), Ultrasonics (often referred to as ultrasonic testing, UT), eddy current testing (ECT), magnetic particle testing (MPIT), and dye penetrant testing (PT). (The American Society of Nondestructive Testing (ASNT) sponsors and has professional level courses in each of these NDT methods for individuals who wish to become "inspectors" with different levels of expertise)

Historically, the primary application of NDE has been to metals. There are few NDE databases for ceramic materials - either monolithics or composites. The metals applications have largely been driven by the inspection requirements for nuclear power plants (mainly stainless steel weldments), coal-fired energy systems (mainly carbon steel pressure vessels), and petroleum refinery pressure vessels and storage tanks as well as other pressure vessel applications. Standard inspection methods for the various components and different applications usually specify the level of a certified inspector required to fulfill that inspection. Insurance companies or warranties by the producer of the component often drives the inspection requirement level. For ceramic materials, at the present time, there are no generally recognized codes or standards. Thus inspection requirements for advanced ceramics are not well defined.

### 3.7.1 Needs and requirements

There are four basic NDE/NDC needs/requirements areas:

- No-Go/Go for accept/reject of as-produced components.
- In-service inspection for replace/continue operations decisions.
- Assessment of degree/extent of in-service damage.
- Determination of quality of damaged component repair.

These four areas apply to stand-alone components and to joints between components.

In the case of CMC materials, for accept/reject for a specific application, the buyer must have established appropriate microstructure requirements. Examples of composite microstructure requirements are:

- The acceptable level and variability of porosity.
- acceptable level and variability of density.
- Required thermal properties.
- Size and extent of acceptable composites "defects", such as delaminations, degree and extent of voids, missing plies, ply drop-offs, and the extent of machining damage.

In-service NDC must be able to detect degree and extent of degradation such as:

- Oxidation of fiber-matrix interface to the extent that such damage would degrade performance.
- On-set and extent of delaminations.
- General degradation such as overall reduced load carrying capacity (for components under load).

It is to be noted in the above comments, that little notice has been given to whether the component has been fabricated using 2D weave lay-ups or 3D braided structures. There is no database currently, with the exception of delamination detection, to allow differentiation between 2D lay-ups and 3D braided structures from an NDE point of view. While the fiber architecture impacts the protocols used and the signal/noise ratios in the final data, fundamentally, there is no significant difference at this time in the application of the NDE/NDC methods.

For the case of joined components, at the current time there is little information at all. What is known is that for brazed ceramic-ceramic joints, the joint needs to be inspected for:

- Completeness of penetration of the braze,
- Uniformity of the braze penetration, and
- Quality of the interface joint strength.

For NDE/NDC needs/requirements for damage standards, little is currently known about how different levels of damage affect the mechanical performance in specific CMC systems. Once this is at least initially assessed, then the requirements for NDC/NDE can be better determined. Using what is now known for 2D lay-up fiber architectures CMCs at the present time, NDE/NDC methods now under development (see subsequent sections) have been shown to detect impact damage such as one might expect from foreign object damage (FOD).

### **3.7.2 Cost**

At the present time, since the NDE/NDC methods have not been totally developed for CMCs, there has not been any major attempts to transfer NDE CMC technology to the private industry segment that sells, maintains, and trains for NDE/NDC equipment. Thus, currently, costs of implementing NDE/NDC methods are not generally established. It is clear is that any NDE/NDC method used must have less than a 5-10% value added cost per component for accept/reject situations.

### **3.7.3 Standards**

For NDE/NDC data to be quantifiable, standards must be developed. Because of differences in density, thermal properties, and modulus in different CFCC systems, standards are necessary for each material system. Therefore, a significant effort should begin to develop these standards and a part of any contractual work should include the development of NDE/NDC test standards. Standards for thickness, density, porosity, void size, and extent for size and location of delaminations are needed for single components. In joints standards are needed for each joint type whether that is a buttjoint, lap joint, or beveled joint. Further, standards are needed for each set of materials joined.

### 3.7.4 Current methods and status

This section is organized, not by the NDE/NDC method, but by the ceramic characteristics/defects. The NDE/NDC method which can detect/measure that parameter is then discussed and the status is given. Currently, there are three (3) basic NDE/NDC methods which have been recently shown to provide significant information in evaluating CMC materials systems. These methods are:

- A. High sensitivity thermal imaging (with 10-12 bit focal plane array CCD detectors) and special computer software.
- B. X-ray imaging (mainly computed Tomographic imaging).
- C. Air-coupled ultrasonic methods (especially with 8-bit dynamic range).

It is to be noted that each of these three methods has been applied to:

- SiC/SiC materials systems with both 2D and 3D fiber architectures (using both CG-Nicalon and Hi-Nicalon) as well as with CVI, PIP, and melt Infiltration processes.
- Al<sub>2</sub>O<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> with sol-gel processing and with filament winding/cloth lay-up fiber architectures.
- SiC/Al<sub>2</sub>O<sub>3</sub> systems with sol-gel processing.
- Joined SiC/SiC materials.

Each of these methods will now be discussed relative to the parameter/characteristic to be detected/measured.

#### 3.7.4.1 Porosity

There are two types of porosity to be detected/measured by NDE/NDC methods: a) distributed open connected to the surface porosity and b) distributed interior porosity not connected to the surface. In NDE/NDC evaluations, porosity in a distributed sense must be somehow separated from single large voids. We define single large voids as defects--not a distributed material characteristic. The amount of distributed porosity is usually determined by CMC fabricators by weigh and measure methods. This yields average bulk porosity; sometimes by determining amount of alcohol infusion (assumes that the porosity is always connected and connected to the surface). This also provides bulk average porosity. Neither of these methods provide an indication of the spatial distribution of porosity.

There are three NDE/NDC methods which can be used to provide a measure of porosity and two of these also provide that information with spatial distribution.

- Thermal imaging with image processing
- Ultrasonic with image processing
- Impact acoustic resonance (no spatial distribution)

##### 3.7.4.1.1 High sensitivity thermal imaging

In high sensitivity thermal imaging, a thermal excitation pulse (using a flash lamp) is provided to the specimen under study and the resulting thermal response from the specimen is time-sequentially recorded by a high sensitivity infrared camera coupled to a computer for data acquisition and data analysis. The result is a measure of thermal diffusivity which can be related to extent of porosity providing that there has been some level of calibration. ASTM has standardized a version of this method and commercially available devices using a laser flash for thermal pulse and limited view detectors are being built and sold. The problem with the commercially available devices is that they are restricted to use of small 1-1 1/2 cm diameter button sized specimens and thus are not useful for full-sized commercial components.

##### 3.7.4.1.1.1 Status

There are two protocols to be used with these systems: a) through transmission where the thermal excitation pulse is on one side of the object and the detector is on the opposite side of the object and b)

single sided where the thermal excitation pulse and the detector are both placed on the same side of the object. The classic case and the one approved by ASTM is the through transmission method. However, because of complex shapes and other limitations, there is a need for one-sided data acquisition. Argonne National Laboratory and others have developed a complete through transmission system which has been applied to full scale components ranging in size up to 30 inches. The limit has been on wall thickness. All components have had wall thicknesses of less than 7 mm with the single exception of a carbon-carbon brake system which had a wall thickness of 25 mm. Another limitation is material system dependence--that is oxide-based materials are optically translucent and thus one needs to use some acceptable method to obtain an optically opaque surface on the thermal impulse side. On turbine engine combustors, where burn-off runs can be used, thin graphite "paints" have been successfully used. Another option is to "tune" the optical spectrum of the thermal excitation pulse so that there is no in-body absorption of the light. This requires knowing the optical transmission properties of the CMC materials.

What is not totally developed at this time is the one-sided capability. Problems with proper algorithms to analyze the data and proper understanding of the thermal pulse details have yet to be fully developed.

#### *3.7.4.1.2 Ultrasonics with image processing*

For ultrasonics with image processing, two ultrasonic transducers are used. One provides an acoustic pulse which "inserts" an elastic wave into the materials, and the second transducer with special low-noise pre-amplifier "receives" the acoustic response from the material under study. As in classic ultrasonic inspections, by scanning the two transducers in a raster pattern over a specimen, an "image" of the acoustic signal is recorded and the strength of that acoustic signal is proportional to the properties of the materials--one of those properties is porosity as porosity attenuates the elastic pulse propagation.

For many CMC systems that have some moisture sensitivity, there is a desire to not insert these materials into water baths which is the most common method of ultrasonic testing. Over the past five years, new technology has allowed the development of air-coupled ultrasonics which does not need any coupling liquid. If the materials of interest were prepared with a range of known porosities, then the ultrasonic attenuation as a function of porosity for that specific material could be established and a calibration would have been developed. In the case of PIP materials this has been done.

#### *3.7.4.1.2.1 Status*

There are two protocols to be used with this method: a) through transmission where the two transducers are placed on opposite sides of the component under study and b) single-sided (so-called Pitch-catch) where the two transducers are placed on the same side of the component under study. The more classic case of ultrasound, where only one transducer is used in a so-called pulse-echo mode, is not valid for CVI, PIP, and Sol-Gel processed CMC materials because these materials have high attenuations as far as sound is concerned. Because of the high attenuation in most CMCs, the center frequency of the transducers used is relatively low, 400kHz. In addition, the transducers used are "focused". That means that the energy of the "sending" transducer is "focused" on the specimen in a fairly small region, about 1 mm square, and the "receiving" transducer is focused to "receive" energy from a fairly small region, also about 1 mm.

For Melt Infiltrated (MI) SiC CMC materials, immersion in water baths present little if any problem and it may well be that the classic pulse-echo method for ultrasound may work. In such a case, the center frequency may also be increased.

In any of these UTS protocols, at this time there is no capability to do contour following and this is badly needed. This is especially true for the one-sided applications where complex shaped objects do not allow through transmission type applications. There also are unresolved issues about the attenuation of CVI, PIP, and Sol-Gel processed materials relative to attenuation as a function of extent of porosity.

### 3.7.4.1.3 Impact acoustic resonance (“The Ping Test”)

For impact acoustic resonance, a transient impulse is imparted to the specimen under study and the resulting vibrations in the component are detected by a non-contact high sensitivity capacitive microphone. There is an ASTM standard method for this test (C 1259- Dynamic Young’s Modulus, Shear Modulus, and Poisson’s Ratio for Advanced Ceramics by Impulse Excitation of Vibration). The J.W. Lemmens Company manufactures an instrument (GrindO-Sonic) which uses the impulse method for determining the elastic modulus of standard flex bars. Argonne National Laboratory (ANL) and others have developed a similar system but for use on larger full scale components. The impact is applied by a controlled electrodynamic shaker with an instrumented “impact” hammer. The sound/vibration is detected by a high sensitivity capacitance microphone. Both of the analog signals are digitized in a high frequency digitizer and the resulting digitized signals are analyzed by frequency analysis software packages. By study of the main acoustic resonance peaks, the specific damping capacity of the specimen can be established, as well as determination of the amplitude of the main resonant peak. Using these two pieces of information, correlations between damping capacity and peak amplitude have been related to porosity. The porosity measurement in this case is an average porosity over the entire component.

#### 3.7.4.1.3.1 Status

ASTM has developed a standard test method for use of impact acoustic resonance for determination of elastic modulus of standard flex bar configurations, assuming isotropic properties. Argonne National Laboratory (ANL) and others have been trying to develop such systems for larger more complex shapes. At issue is the need to verify the vibratory mode shapes developing under the impact load such that the vibratory analytical models are verified and thus the appropriate detected frequency is used in the analysis. For complex shaped structures with complex vibratory patterns this analysis is extremely intricate. One possible solution, currently being approached, is to use new scanning laser vibrometers with a very high frequency response, coupled to new analytical software packages that allow detailed vibratory modes to be established. If this would work, then indeed an indication of the spatial distributions of porosity could be established by this method as well as establishing the reliability of this method for global determination of porosity.

#### 3.7.4.2 Density

There are two density measurements of interest: a) bulk density and b) density distribution (perhaps even in 3-D). Density is usually measured by CMC fabricators using the Archimedes methods with some kind of sealant to seal off water (or other fluid) impregnation. However, this calculated porosity value is a bulk value. There is a need to know the spatial distribution of porosity, since the analysis of CMC component performance by finite element analysis requires knowledge of the density and porosity distribution through the component.

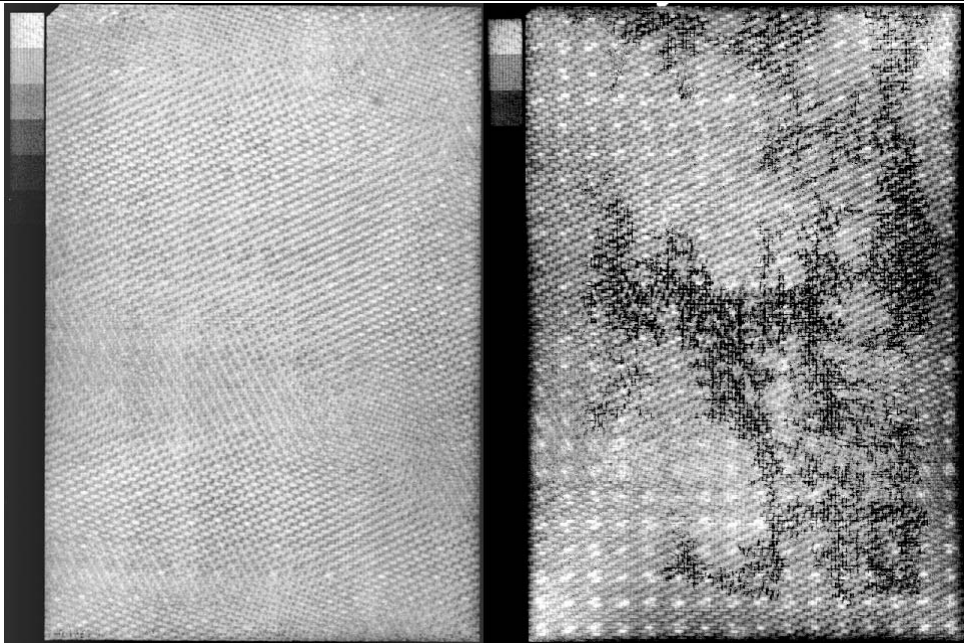
There are two primary NDE/NDC methods to measure density. Each of these methods has the capability to provide the information in 3-D mapping: a) X-ray imaging (primarily Tomographic) b) Ultrasonic Methods.

##### 3.7.4.2.1 X-ray imaging

###### 3.7.4.2.1.1 X-ray radiography

Shown in Figure 3.7.4.2.1.1 are two conventional radiography images taken from two fully processed Sylramic™/SiC/MI panels. Both panels have an overall dimension of six inches by nine inches and a nominal thickness of 0.090 inches. The grayscale levels shown in these two images can be correlated directly with through-thickness density. For this purpose, a 1/2-inch by 2 1/2-inch radiographic calibration standard is positioned in the upper left-hand corner of each panel image. The presence of the witness sample provides a consistent calibration standard that is used to quantitatively assess the relative density differences between separate panel radiographs. The witness sample is the same Sylramic™/SiC/MI material in a fully dense state. The lightest square in the witness sample radiograph has the same nomi-

nal thickness as the panel. The remaining length of the witness sample has sequential 0.005-inch steps machined into the thickness. These steps provide a quantitative method for converting the grayscale level in a panel image to a density equivalent. Since both panels have an equivalent and constant thickness, any variation in the grayscale levels are the result of variations in the through-thickness density. The radiographic image from panel 1 has the uniform appearance associated with what is considered a good quality panel based on the NDE technique employed. The artifacts in the radiographic image of panel 2 are typically associated with a lower quality panel. The large dark regions visible in the image for panel 2 are the result of reductions in the through-thickness density induced by areas of entrained porosity.



**FIGURE 3.7.4.2.1.1** *X-ray radiograph taken through a “good” and “bad” composite sample on the left and right, respectively.*

#### 3.7.4.2.1.2 *X-ray computed tomography*

In x-ray computed tomography, an X-ray source and an X-ray detector are coupled (shown is a so-called 2-Dimensional or area detector). The output of the detector is coupled directly to a very powerful PC or workstation loaded with sophisticated image processing software. The object under study is placed on a rotatable table, controlled by the computer, between the X-ray source and the detector.

For simple projection X-ray imaging, the strength of the X-rays from the X-ray source is such that the X-rays penetrate the object and a shadowgraph type image appears on the computer screen. In cases where the component is a flat or nearly flat plate, then indeed by projecting the image of the component onto the X-ray detector, a relative correlation can be obtained between density and the gray tones in the presented image. There would be, however, no distribution of density through the thickness of the plate.

For density mappings of objects of more complexity than simple flat plates, it is necessary to use computed tomographic imaging. In this case, it is necessary to obtain a sequence of projection images using the computer controlled table. The set of images (really X-ray attenuation data) are then used to “reconstruct” cross sections of the object. Because the reconstructed image is obtained from measurements of the relative X-ray attenuation, if properly obtained, the image gray tones can be correlated to density; and since a 3-D image can be obtained, the density can be determined in 3-D (the use of X-ray imaging to map density variations is known as X-ray densitometry).

ASTM has also developed guides for X-ray computed tomography (XCT) and now is developing guides for densitometry applications. Spatial resolution achievable in either method depends upon many variables. These include the size of the object under study, the specifics of the detector used, the elegance of the software and how careful the calibration of the device was done.

#### 3.7.4.2.1.2.1 Status

There are two protocols to be used for X-ray imaging: a) through transmission projection imaging and b) computed tomographic imaging (often called CAT scanning). As noted earlier, ASTM has developed guides for X-ray imaging and recently has developed guides for assessing image quality of X-ray imaging devices as well as guides for using CAT scanner for densitometry studies. For CMC materials, there is a need for standards for the densitometry studies and also for very high speed computer codes, as currently the data acquisition times and image processing times are still too high.

#### 3.7.4.2.2 Ultrasonics with image processing

The value of ultrasonic imaging can be vastly extended through the use of data processing to analyze the image information. What is important to state here is that elastic modulus is directly related to density. If a set of specimens with variations in density alone were developed for all CMC materials, then indeed ultrasonic attenuation as a function of density could be realized. Through-thickness density analysis is much more complex and there would need to be a very sophisticated software program developed to through-the-depth profiling as a function of frequency and then extract the attenuation to get 3-D density.

#### 3.7.4.3 Required thermal properties

CMC materials are usually applied in high temperature environments. Because of this, the distribution of thermal properties on a single component is very much of interest. Most CMC fabricators today use the ASTM standard test for measuring thermal properties (thermal conductivity, thermal diffusivity) on specimens to develop the process. As noted earlier, ANL has now developed NDC/NDE systems that allow full field mapping of thermal properties (diffusivity) on full scale components. Care must be exercised between oxides and non-oxides as noted earlier. Oxides are partially translucent in the infrared and optical spectrum and care needs to be exercised when data are obtained using infrared methods.

There is only one NDE/NDC method to measure thermal properties -- infrared imaging. This method and the current status was described earlier.

#### 3.7.4.4 Defect detection

Defects in ceramic matrix composites can be categorized as a) delaminations, b) degree and extent of voids, c) missing plies and ply drop-offs, and d) extent of machining induced damage.

##### 3.7.4.4.1 Delaminations

Delaminations occur mainly in 2D cloth lay-up fiber architecture and to lesser extents in filament wound fiber architectures. Because delaminations occur parallel to the surface, through transmission X-ray imaging does not detect these defects. However, since delaminations effect heat transfer, as well as act as significant attenuators to elastic wave propagation, both infrared imaging and ultrasonic methods can detect delaminations. For complex shaped structures, X-ray CAT scanning is useful and has been shown to be able to detect delaminations. There is no part size limitations to any of these methods.

Quantification of delaminations is interpreted to mean size and depth-location. By using one-sided thermal imaging, again if a calibration standard is available, the depth of the delamination can be determined. The same approach is applicable to air-coupled or water-coupled ultrasonic systems. X-ray CAT scans provide depth-of-delamination almost by definition.



#### 3.7.4.4.2 Degree and extent of voids

Large voids (usually considered to be greater than 1-2 mm in maximum dimension) can usually be detected in flat plate shaped specimens by through transmission X-ray imaging (provided the specimen thickness is not too great (i.e., less than 6 mm thick)). For complex shaped specimens, such large voids have been demonstrated to be detected by X-ray CAT scanning techniques. While infrared and ultrasound would seem to be alternative methods, heat diffusion and scattering of ultrasound, while allowing detection, do not lend themselves to the direct measurement of voids in the way that the X-ray imaging systems do.

#### 3.7.4.4.3 Missing plies

In complex shaped structures where ply lay-ups are necessary for added strength through the addition of ply layers, the ability to determine whether or not a ply layer is missing is important. Depending upon the local geometry (i.e., whether there is significant curvature), experience has shown that thermal imaging using the new 12-bit dynamic range cameras with the proper band pass lens is so sensitive that the existence of the necessary ply layers can be detected. If the geometry is very complex, then X-ray CAT scanning may be employed but this has so far not been necessary.

#### 3.7.4.4.4 Ply drop-offs

When fabricating a complex shaped CMC structure using 2D fiber architectures, there are certain situations in the lay up of the fabric, where there can be a tendency for ply layers to shift in one direction or another during fabrication. What this results in is the situation where ply layers do not go all the way to some desired boundary and you get so-called ply drop-off. From an NDE/NDC point of view this is analogous to missing plies and the same logic applies.

#### 3.7.4.4.5 Machining induced damage

Depending on the damage tolerance of the CMC, machining damage/defects may or may not be critical flaws with a direct impact on performance. However, in the majority of cases, machining induced damage is a surface flaw which can be evaluated by optical or dye penetrant methods, rather than with the more complex ultrasonic, X-ray, and thermal imaging methods.

#### 3.7.4.5 In-service inspection (ISI) by NDE/NDC

In-service inspection (ISI) will be defined as that inspection procedure (NDC/NDE) such that components can be assessed for their status in use. With ISI information, critical decisions can be made whether or not to continue to operate with that component or to remove and replace the component. For man-rated applications or for systems which cost down-time, the "replace" decision can be costly/risky, and the wrong answer can result in severe consequences.

For CMC materials, environmental and cumulative effects of concern are:

- Oxidation of the fiber-matrix interface with known oxidation susceptibility such as pyrolytic carbon (PC) or Boron Nitride (BN)
- Foreign object damage
- Formation of delaminations
- For CMC joined materials there will be additional concerns as yet unspecified

##### 3.7.4.5.1 Fiber-matrix interface degradation

While far from being proven at the moment, there are two main candidate NDE/NDC methods with the potential to detect this type of material degradation: a) impact acoustic resonance, because the change in local clamping stresses caused by the degradation results in changes in damping capacity as well as changes in the overall structural stiffness, producing a drop in natural frequency; and b) thermal

imaging, because the change in fiber-matrix coating changes the thermal conductivity of the material. Both of these methods require further development and verification.

#### *3.7.4.5.2 Foreign object damage (FOD) and delaminations*

This is a new area under study from a CMC point of view and little NDE/NDC data are available. However, FOD tends to resemble delaminations with extended damage regions. To date, thermal imaging, air-coupled Ultrasonic and X-ray imaging have shown promise for detecting and quantifying the damage region.

### **3.7.5 Developing methods**

Reserved for future use.

## **3.8 QUALITY CONTROL OF STARTING MATERIALS**

Reserved for future use.

## **3.9 MACHINING**

Reserved for future use.

## **4 QUALITY CONTROL OF FINAL PRODUCTS**

Reserved for future use.

### **4.1 INTRODUCTION**

Reserved for future use.

### **4.2 QUALITY ASSURANCE**

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### **4.3 MATERIAL PROPERTY VERIFICATION**

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### **4.4 STATISTICAL PROCESS CONTROL**

Reserved for future use.

## **5 APPLICATIONS, CASE HISTORIES AND LESSONS LEARNS**

Reserved for future use.

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HANDBOOK**

**CERAMIC MATRIX COMPOSITES**

**VOLUME 5**

**PART B. DESIGN AND SUPPORTABILITY**

## **PART B. DESIGN AND SUPPORTABILITY**

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**DEPARTMENT OF DEFENSE  
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**CERAMIC MATRIX COMPOSITES**

**VOLUME 5**

**PART C. TESTING**

## PART C. TESTING

### 8 THERMO-MECHANICAL-PHYSICAL TEST METHODS - OVERVIEW

#### 8.1 INTRODUCTION

This chapter provides guidelines for the experimental thermo-mechanical-physical characterization and its subsequent documentation for ceramic matrix composites and their constituents within MIL-HDBK-17. Recommended standardized test methods, test parameters, and test matrices for a number of uses are presented and discussed.

##### 8.1.1 Building block approach

Analysis alone is generally not considered adequate for substantiation of composite structural designs. Instead, the “building-block” approach to design development testing is used in concert with analysis. This approach is often considered essential to the qualification/certification of composite structures because of the sensitivity of composites to out-of-plane loads, multiplicity of failure modes, environmental influences, and the lack of widely accepted/applicable analytical models/methodologies.

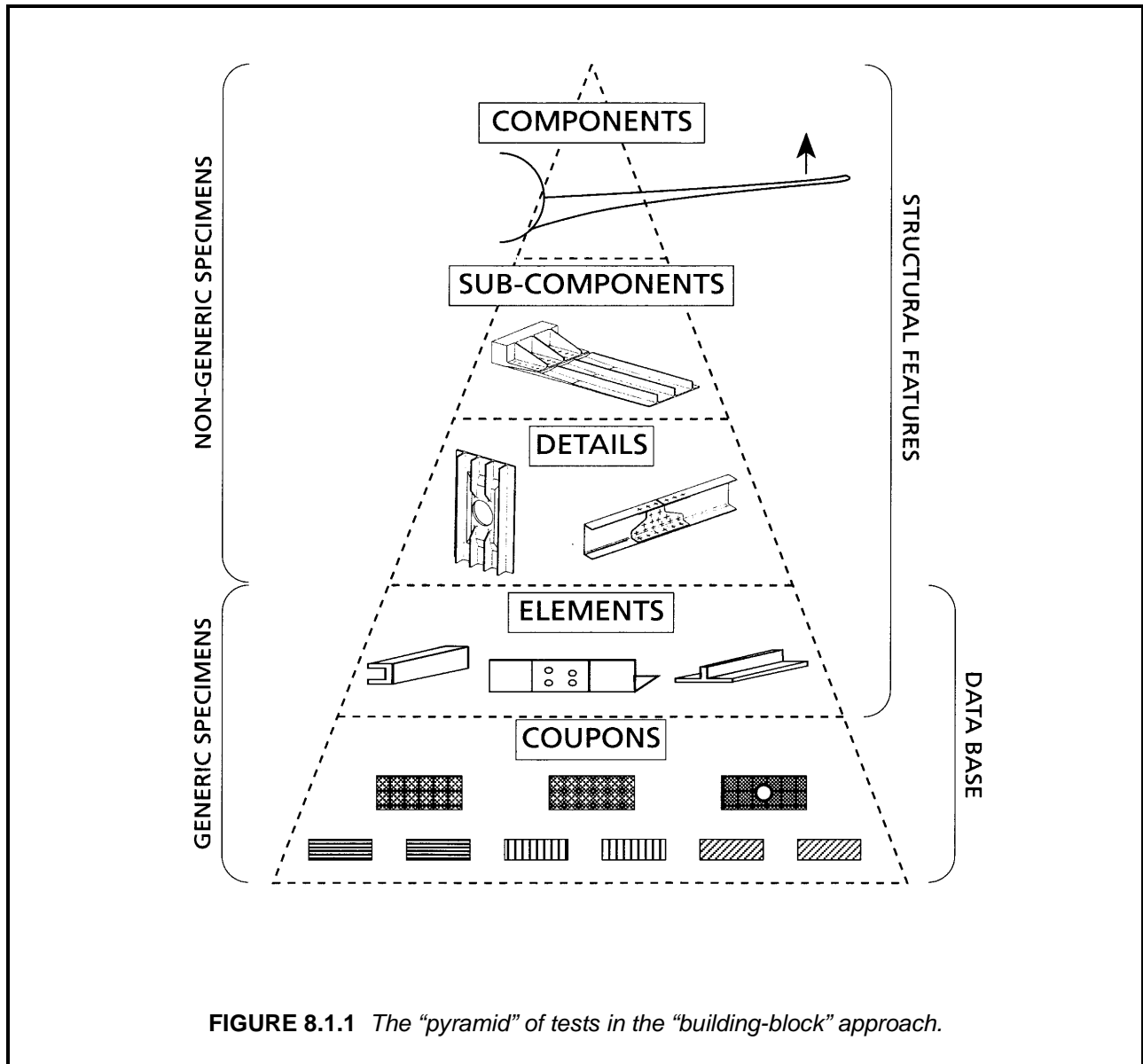
The building-block approach may be used to establish time-dependent environmental compensation values applied to full-scale tests under ambient conditions, as it is often impractical to conduct these tests under the actual environment, moisture, and temperature of the intended use. Lower-level tests may justify these environmental compensation factors. Similarly other building block tests may determine truncation approaches for cyclic mechanical fatigue, and cyclic thermal fatigue as well as compensation for fatigue scatter at the full scale level.

The building block approach is shown schematically in Figure 8.1.1 and may be summarized in the following steps:

1. Generate material basis values and preliminary design allowables.
2. Based on the design/analysis of the structure, select critical areas for subsequent test verification.
3. Determine the most strength-critical failure mode for each design feature.
4. Select the test environment that will produce the strength-critical failure mode. Special attention should be given to constituent-sensitive failure modes (such as oxidation of interphase, recession of matrix, or degradation of fiber) and potential “hot-spots” caused by out-of-plane forces or stiffness tailored designs.
5. Design and test a series of test specimens, each one of which simulates a single selected failure mode and loading condition, compare to analytical predictions and adjust analysis models or design allowable as necessary.
6. Design and conduct increasingly more complicated tests that evaluate more complicated loading scenarios with the possibility of failure from several potential failure modes. Compare to analytical predictions and adjust analysis models as necessary.
7. Design (including compensation factors, if necessary) and conduct, as required, full-scale component static and fatigue testing for final validation of internal forces and structural integrity. Compare to analysis.

##### 8.1.2 Test level and data uses

Testing activities can be defined in two basic ways: Structural Complexity Level and Data Application Category. The classes within each are discussed in more detail in the following sections and can be used to map large-scale testing programs to aid in test planning.



#### 8.1.2.1 Structural complexity levels

The five structural complexity levels are each geometry or form based: constituent, mini-composite, bulk-composite, structural element and structural subcomponent. The material form(s) to be tested and the relative emphasis place on each level, should be determined early in the material data development planning process and will likely depend on many factors, including: manufacturing process, structural application, corporate/organizational practices, and/or the procurement or certification agency. While a single level may suffice in rare instances, most applications require at least two levels and it is common to use all five in a complete implementation of the building-block approach. Regardless of the structural complexity level selected, physical, chemical, and processing aspects of the fiber, interphase, matrix are necessary to support thermo-mechanical-physical property test results. Each procurement or certification agency has specific minimum requirements and guidelines for use of data. Users of MIL-HDBK-17 are advised to coordinate with the procuring or certifying agency before planning and conducting any testing that supports structural qualification or certification.

The five structural complexity levels cover the following areas:

*Constituent Testing* – This evaluates the individual properties of fibers, fiber forms, interphases, matrices, and overcoats. Key properties may include density, strength, or stiffness.

*Mini-Composite Testing* - This evaluates the properties of the fiber, interphase, and matrix in the composite material form, but often limited to single yarn rather than lamina of fabric. Key properties may include matrix cracking, fiber debonding, environmental effects.

*Bulk-Composite Testing* - This evaluates the response of the composite material in a given lay-up (stacked textile plies, or stacking unidirectional laminas, etc.) Key properties include proportional limit behavior, ultimate strength, elastic constants.

*Structural Element Testing* - This evaluates the ability of the material to tolerate common bulk composite discontinuities. Key properties include open hole tension/compression, notched tension/compression, joint shear/bearing and interlaminar response.

*Structural Subcomponent (or higher) Testing* - This testing evaluates the behavior and failure mode of increasingly more complex structural elements. These are application specific and are not specifically covered in MIL-HDBK-17.

#### 8.1.2.2 Data application categories

Material property testing can also be grouped by data application into one or more of the following four categories: screening, qualification, acceptance and equivalence. The starting point for testing most material systems is usually material screening. Material systems intended for used in engineering hardware are subjected to further screening to obtain additional data. The four data application categories are described as follows.

##### 8.1.2.2.1 Screening tests

This is the assessment of candidate materials for a given application, often with particular application/environment/component in mind. The purpose of screening is initial evaluation of new material systems under worst case environmental and loading test conditions. Guidelines for these so-called “killer tests” are provided in MIL-HDBK-17. The MIL-HDBK-17 screening test matrix provides values for various strength, moduli and physical properties and is designed to eliminate deficient material systems from the material selection process and to reveal promising new material systems before planning subsequent, more in-depth, evaluations.

##### 8.1.2.2.2 Material qualification tests

This step proves the ability of a given material/process to meet the requirements of a material specification. It is also the process of establishing the original specification requirement values. Rigorous material qualification testing considers the statistics of the data and is ideally a subset of, or directly related to, the design allowable testing performed to satisfy structural substantiation requirements. (However, while a material may be qualified to a given specification, it still must be approved for use in each specific application.) The objective is quantitative assessment of the variability of key material properties leading to various statistics that are used to establish material acceptance, equivalence, quality control, and design basis values. Since there are various sampling and statistical approaches used within the industry, the approach used must be explicitly defined. While a generic B-basis value can be obtained many ways, a fully-approved MIL-HDBK-17 B-basis value carries with it a specific sampling and statistical determination process and emphasizes additional considerations like test methodology, failure mode and data documentation.

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*8.1.2.2.3 Acceptance tests*

This is the task of verifying material consistency through periodic sampling of material product and evaluation of key material properties. Test results from small sample sizes are statistically compared with control values established from prior testing to determine whether or not the material production process has changed significantly.

*8.1.2.2.4 Equivalence tests*

This task assesses the equivalence of an alternate material to a previously characterized material, often for the purposes of utilizing an existing material property data base. The objective is evaluation of key properties for test populations large enough to provide a definitive conclusion, but small enough to provide significant cost savings as compared to generating an entirely new data base. A significant use includes evaluation of possible second source of supply for a previously qualified material. However the most common uses of this process are: 1) evaluation of minor constituent, constituent processing or fabrication processing changes for a qualified material system; and 2) substantiation of previously established MIL-HDBK-17 basis values.

**8.2 TEST PROGRAM PLANNING****8.2.1 Overview**

A matrix is shown in Table 8.2.1 that can be used in test planning for large-scale testing programs. The material property tests from structural complexity levels and data application categories are listed on the axes of the array with each intersecting cell describing a distinct testing activity (though certain combinations will rarely be used). Groups of cells can be used to summarize the scope of entire building block testing programs. The array shown in Table 8.2.1 illustrates a common (but by no means universal) testing sequence in the substantiation of a composite-based aerospace structural application. The sequence begins with the cells at the upper left of the array and proceeds with time, toward the cell at the lower right with the numbered notes indicating the approximate order of the sequence.

**TABLE 8.2.1** *Test program definition.*

Structural Complexity Level	Data		Application	Categories
	Material Screening	Material Qualification	Material Acceptance	Material Equivalence
Constituent	1	-	-	
Mini Composite	2	4		
Bulk Composite	-	5		7
Structural Element	3	6		8
Structural Subcomponent	-			9



**8.2.2 Baseline and alternate approaches for statistically-based properties**

Reserved for future use.

**8.2.3 Issues of data equivalence**

Reserved for future use.

**8.2.4 Test method selection**

Reserved for future use.

**8.2.5 Population sampling and sizing**

Reserved for future use.

**8.2.6 Material and processing variation**

Reserved for future use.

**8.2.7 Material operating limit**

Reserved for future use.

**8.2.8 Non ambient testing**

Reserved for future use.

**8.2.9 Data normalization**

Reserved for future use.

**8.2.10 Data documentation**

Reserved for future use.

**8.2.11 Application specific testing needs**

Reserved for future use.

**8.3 RECOMMENDED TEST MATRICES****8.3.1 Material screening**

The objective of the screening process is to reveal attributes and/or inadequacies of key properties and performance of new candidate material systems, while keeping testing to a minimum. The screening process identifies, for a particular composite material system, the critical test and environmental conditions as well as any other special considerations. Proper test matrix design enables comparison with current production material systems.

The general approach to design of a screening test matrix is selection of key static tests that provide sufficient data to assess mean values of stiffness and strength at both mini and bulk composite levels. The mini composite level tests provide intrinsic material stiffness and strength properties commonly used in ceramic composite micro mechanical models including tension, compression and shear loading. The bulk composite level tests provide screening strength data on application issues relating to stress discon-

tinuities, such as transition radii and notches. Baseline tests are performed under ambient conditions. However, “killer tests” are performed under critical controlled environment conditions in order to quickly reveal inadequacies of the material system.

An example of a mechanical property screening test matrix is shown in Table 8.3.1. Under extreme environments, additional factors may have to be considered as discussed in subsequent sections. Sensitivity to exposure of operational conditions and other special issues may justify additional special tests in the screening evaluation.

**TABLE 8.3.1** *Example of a material screening matrix for static strength.*

Test	Number of Test Specimens		Evaluation Emphasis
	Room Temp. Ambient	High Temp Environment	
Mini Composite:			
Tension	3	3	fiber
Compression	3	3	fiber/matrix
Shear	3	3	interphase
Bulk Composite:			
Open Hole Compression	3	3	stress raiser
Compression after Impact	3	3	damage
Notched Tension	3	3	stress riser

### 8.3.2 Material qualification

Recommended thermal, mechanical, and physical property test matrices are based on replicate tests conducted across batches of materials (see Tables 8.3.2(a) and (b)). For thermal and physical properties, at least three replicate tests are to be conducted across at least five batches to provide for the parametric/nonparametric analysis when determining B-basis properties. For mechanical properties, at least six replicate tests are to be conducted across at least five batches to provide for the parametric/nonparametric analysis when determining B-basis properties. Fewer replicates or batches may be acceptable if agreed to between contractor and the procuring agency.

### 8.3.3 Material acceptance test matrices

Reserved for future use.

### 8.3.4 Alternate material equivalence test matrices

Reserved for future use.

**TABLE 8.3.2(a)** *Example of a material qualification matrix for thermal/physical properties.*

Thermal/Physical Property	Test Methods	Number of Tests per Batch for each Test Condition	Number of Tests per Batch for each Test Condition	Number of Tests
	See Handbook Section	Room Temp. Ambient	High Temp Environment	
Fiber Volume	7.5.4.3	3	N/A	15
Matrix Volume	7.5.4.3	3	N/A	15
Density	7.5.4.3	3	N/A	15
Diffusivity	7.5.4.1	3	N/A	15
Expansion	7.5.4.1	3	N/A	15
Specific Heat	7.5.4.1	3	N/A	15

**TABLE 8.3.2(b)** *Example of a material qualification matrix for mechanical properties.*

Mechanical Property	Test Methods	Number of Tests per Batch for each Test Condition	Number of Tests per Batch for each Test Condition	Number of Tests
	See Handbook Section	Room Temp. Ambient	High Temp Environment	
In-plane Tension	9.4.2	6	6	60
Trans-thickness Tension	9.4.2	6	6	60
Compression	9.4.2	6	6	60
In-plane Shear	9.4.2	6	6	60
Interlaminar Shear	9.4.2	6	6	60

**8.3.5 Generic material/structural element test matrices**

Reserved for future use.

**8.3.6 Alternate approaches to basis values**

Reserved for future use.

**8.3.7 Data substantiation for use of MIL-HDBK-17 basis values**

Reserved for future use.

## 8.4 DATA REDUCTION AND DOCUMENTATION

### 8.4.1 Introduction

Reserved for future use.

### 8.4.2 Layer properties from composites

Reserved for future use.

### 8.4.3 Data normalization

Data analysis is performed on thermal, physical and mechanical test data for a variety of reasons that include determination of multi-batch statistics and statistically-based property values (allowables), comparison of materials from different sources, material selection, evaluation of processing parameters and quality assurance evaluation. Such calculations or direct comparisons may not be valid if test specimens with different fiber volume contents are tested. Normalization is a procedure for adjusting raw test values to a single (specified) fiber volume content. The following subsections discuss the theory, methodology and practical application of normalization.

#### 8.4.3.1 Normalization theory

Properties that are dominated by the properties of the reinforcing fibers are dependent on the volume fraction of the fiber in the composite. For example, in the commonly used “rule of mixture” model, zero degree in-plane tensile strength of a unidirectionally reinforced composite is assumed equal to the matrix tensile strength at 0% fiber volume and equal to the fiber strand tensile strength at 100% fiber volume. Neglecting the effects of porosity, the relationship between fiber volume fraction and ultimate tensile strength is, therefore, linear over the entire range of fiber/matrix ratios. This follows for the fact that volume percent fiber is the same as the area percent fiber in the test specimen cross section. Tensile modulus is expected to follow the same behavior. Thus, test specimens having different volume contents have fiber dominated properties that vary linearly with fiber volume fraction.

Two factors can cause composite fiber volume fraction to vary: 1) the amount of matrix present relative to the amount of fiber (matrix fraction) and 2) the amount of porosity (void volume). These factors give rise to changes in fiber volume fraction from material-to-material, batch-to-batch, panel-to-panel and even test specimen to test specimen within a panel. In order to perform data analysis that compares materials, batches, panels or test specimens, the data for fiber-dominated properties must be adjusted to a common fiber volume fraction. If this is not done, an additional source of variability will be included in the data that might lead to erroneous conclusions. The process of data normalization attempts to remove or reduce this source of variability.

#### 8.4.3.2 Normalization methodology

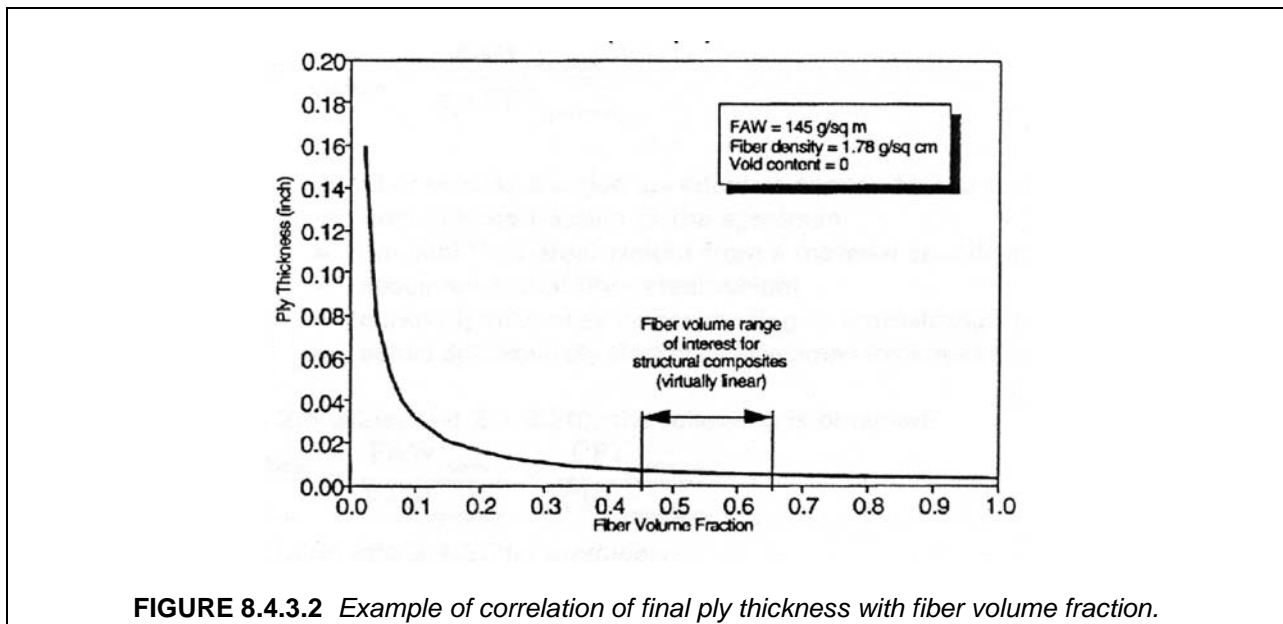
Since, in theory, fiber-dominated strength and stiffness properties vary linearly with fiber volume fraction, an obvious first approach would be to determine the actual fiber volume fractions of the test specimens by an appropriate method and to adjust raw data values by the ratio of a common fiber volume fraction (chosen or specified) to the actuals as shown in Equation 8.4.3.2(a).

$$\text{Normalized value} = \text{Test value} \times \frac{FV_{\text{normalizing}}}{FV_{\text{specimen}}} \quad 8.4.3.2(a)$$

where  $FV_{\text{normalizing}}$  is the chosen fiber content (volume fraction or %) and  $FV_{\text{specimen}}$  is the actual fiber content (volume fraction or %).

Although this would appear to be the most direct approach, it has limitations. The most serious deficiency is that fiber volume is not commonly measured for each individual test specimen. At best, representative pieces from each test panel are used to estimate the average panel fiber volume fraction.

A preferred method of data normalization employs an approach that accounts for the fiber volume variation between individual test specimens. The basis of this method is the relationship between fiber volume fraction and final ply thickness. As stated earlier, fiber volume fraction is a function of matrix content and void content. At a given void content, fiber volume fraction is entirely dependent upon matrix content. Furthermore, for a given void content and fiber areal weight, panel thickness (and hence ply thickness) is also dependent only upon matrix content. Thus, it follows that final ply thickness is solely dependent upon fiber volume fraction for constant fiber areal weight and void content. This dependency permits normalization of each individual test specimen by its ply thickness (total thickness divided by number of plies). An example of this relationship between final ply thickness and fiber volume fraction (which is virtually linear within the 0.45 to 0.65 fiber volume fraction range of usual interest for structural composites) is shown in Figure 8.4.3.2.



**FIGURE 8.4.3.2** Example of correlation of final ply thickness with fiber volume fraction.

A somewhat involved derivation gives a relation for normalizing individual test specimens such that:

$$\text{Normalized value} = \text{Test value} \times \frac{\text{FAW}_{\text{no min al}}}{\text{FAW}_{\text{specimen}}} \times \frac{\text{CPT}_{\text{specimen}}}{\text{CPT}_{\text{normalizing}}} \quad 8.4.3.2(b)$$

where  $\text{FAW}_{\text{no min al}}$  is the nominal fiber areal weight from a material specification or other source,  $\text{FAW}_{\text{specimen}}$  is the test specimen actual fiber areal weight,  $\text{CPT}_{\text{normalizing}}$  is the cured ply thickness corresponding to normalizing fiber volume fraction, and  $\text{CPT}_{\text{specimen}}$  is the cured ply thickness of the test specimen (i.e., test specimen thickness divided by the number of plies).

Equation 8.4.3.2(b) can be transformed to a simpler form if  $\text{CPT}_{\text{normalizing}}$  is not calculated and the assumption is made that the fiber areal weight does not vary greatly within a batch of material. Thus, it can be assumed that  $\text{FAW}_{\text{specimen}}$  can be approximated as the batch fiber areal weight,  $\text{FAW}_{\text{batch}}$ :

$$\text{Normalized value} = \text{Test value} \times \frac{FV_{\text{normalizing}}}{FAW_{\text{batch}}} \times \rho_f \times CPT_{\text{specimen}} \quad 8.4.3.2(c)$$

where  $\rho_f$  is the fiber density (mass/volume).

#### 8.4.3.3 Practical application of normalization theory

Common practice is to normalize fiber-dominated properties (both unnotched and notched for mechanical properties) for composites fabricated from unidirectional laminates, multi-directional fabrics, and rovings (i.e., windings). Although fiber volume effects on various matrix-dominated properties (in-plane and interlaminar shear or trans-thickness tensile, for example) have been observed, there is no clear model for these effects, and such properties are not normalized. Generally in MIL-HDBK-17, normalized values are presented for all mechanical strength and stiffness properties except: 90° (transverse) tension and compression of unidirectional laminates, interlaminar (3- or z-direction) tension, interlaminar shear, in-plane shear, short beam strength, bearing, bearing/bypass, strain energy release rate, and Poisson's ratio.

Laminates fabricated from rovings and similar forms using a winding process present a unique situation relative to normalization. Such constructions do not have plies in the usual sense: the wound "ply" thickness depends upon tow band width, wind spacing, and tow spread during winding. Since nominal ply thickness and fiber areal weight are not directly applicable, normalization by ply thickness and fiber areal weight is not possible. Test data for these materials must be normalized using the ratio of normalizing fiber volume fraction to the average measured panel fiber volume fraction.

When fiber-dominated properties are normalized, data scatter should decrease compared to the unnormalized values since variability due to fiber volume fraction differences is being reduced. Thus, coefficients of variation should be lower after normalization. However, this is not always observed, and there are a number of reasons why the reduction in scatter expected from normalization is not invariably realized:

1. If measured ply thicknesses are close to the normalizing thickness and the fiber areal weight is close to nominal, correction factors will be small, and may be nearly the same magnitude as errors in measuring these quantities.
2. The mode of failure initiation may change as a function of fiber volume. As an example, measured (unnormalized) compression strength may increase as fiber volume fraction increases over a given range. However, at some point additional fiber may not increase strength because the ability of the matrix to support the fibers has been exceeded, and a stability failure occurs on a macro scale. In this case, the relationship between strength and fiber volume breaks down, and data scatter is not necessarily reduced by normalization.
3. Flaws in test specimens might cause premature failures. If some specimens fail because of flaws and others at the true material limit, results of normalization will not be predictable.
4. If the coefficient of variation is already small (less than 3%, for example), further reduction as a result of normalization should not be expected, since this level of variability is about the minimum usually observed for most composite properties.

No change in data scatter after normalization is usually not a cause for concern. However, if data scatter increases significantly after normalization, the reason should be investigated.

#### 8.4.4 Data documentation requirements

Reserved for future use.

## **9 MATERIAL TESTING & CHARACTERIZATION FOR SUBMISSION OF DATA TO MIL-HDBK-17**

### **9.1 INTRODUCTION**

Reserved for future use.

### **9.2 MATERIAL AND PROCESS SPECIFICATION REQUIREMENTS**

Reserved for future use.

### **9.3 DATA SAMPLING REQUIREMENTS**

Reserved for future use.

### **9.4 TEST METHOD REQUIREMENTS**

This section is divided into three major subsections: Thermal, Mechanical, and Physical. Each subsection is further sub-divided into sub subsections for each type of test method. Finally, contained in each sub subsection are references to and summaries of test methods for both bulk CMCs and each of the major constituents: fiber, matrix, interphase, and overcoat. The specific national and international full-consensus test method for each type of test is listed and the test parameters for each test method are prescribed for qualifying materials for inclusion in MIL-HDBK-17.

#### **9.4.1 Thermal**

Thermal test methods include those which relate directly to thermal properties which directly influence a material's design use. In particular, conductivity, diffusivity, expansion, specific heat, thermal shock, and thermal fatigue are highlighted.

##### *9.4.1.1 Conductivity*

There are no national or international standards for thermal conductivity of bulk CMCs or their constituents.

##### *9.4.1.1.1 Bulk CMC*

There are no national or international standards for thermal conductivity of bulk CMCs.

##### *9.4.1.1.2 Matrix*

There are no national or international standards for thermal conductivity of CMC matrices.

##### *9.4.1.1.3 Fiber*

There are no national or international standards for thermal conductivity of CMC fibers.

##### *9.4.1.1.4 Interphase*

There are no national or international standards for thermal conductivity of CMC interphases.

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*9.4.1.1.5 Overcoat*

There are no national or international standards for thermal conductivity of CMC overcoats.

*9.4.1.2 Diffusivity*

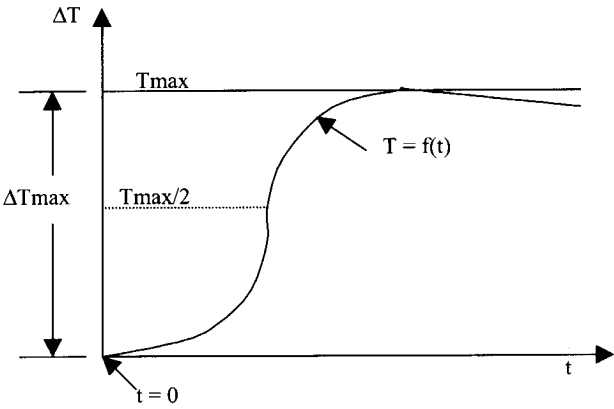
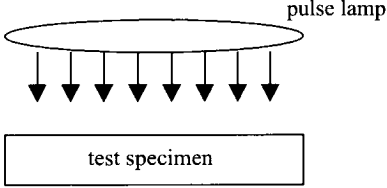
The only national or international standard for thermal diffusivity of CMCs has been developed for bulk CMCs only.

*9.4.1.2.1 Bulk CMC*

The only national or international standard for thermal diffusivity of CMCs has been developed by CEN for bulk CMCs only. An overview of CEN ENV 1159-2 "Thermal Diffusivity of Continuous Fiber Reinforced Ceramic Composites" (see Table 9.4.1.2.1).



**TABLE 9.4.1.2.1** CEN ENV 1159-2 "Thermal Diffusivity of Continuous Fiber-Reinforced Ceramic Composites".

<p><b>1. Outline:</b>                  Object Material: continuous fibre reinforced ceramic matrix composite                  Test Method: environment control chamber                  Test Environment: ambient or inert air                  Number of Specimens: minimum of three test specimens</p>	 <p>Figure 2 Example of thermogram</p>
<p><b>2. Apparatus:</b>                  Heat pulse source (flash tube or pulse laser), environmental control chamber, transient temperature detector (infrared or thermocouple)</p>  <p>Figure 1 Schematic illustration of test set-up</p>	
<p><b>3. Procedure:</b></p> <ul style="list-style-type: none"> <li>- Pulse duration: <math>t_d \leq \frac{0.002e^2}{\alpha}</math> where <math>t_d</math> is response time (s), <math>e</math> is the thickness (m) and <math>\alpha</math> is the thermal diffusivity (<math>m^2/s</math>)</li> <li>- Measure thickness of test specimen with calipers in accordance to ISO 6906</li> <li>- Fix test piece in test position</li> <li>- Start record before pulse to determine baseline</li> <li>- Record thermogram</li> </ul>	<p><b>6. Report:</b></p> <ul style="list-style-type: none"> <li>Name and address of testing establishment</li> <li>Date of test, unique identification of report</li> <li>Customer name, address and signatory</li> <li>Reference to this standard</li> <li>Description of test material, type of fibre, batch number, date of receipt</li> <li>Description of test piece; dimensions, preparation method, orientation, mass, volume</li> <li>Brief details of equipment used</li> <li>Relevant test parameters</li> <li>Transient detector employed</li> <li>Environmental conditions; vacuum, inert gas, etc.</li> <li>Measured values at transient half time</li> <li>Thermogram</li> <li>Biot's number and characteristic time</li> <li>Theoretical calculated curve and residue</li> <li>Calculation method used</li> <li>Results of individual tests on required number of test pieces</li> <li>Mean results and standard deviation</li> <li>Comments about test and/or test results</li> </ul>
<p><b>4. Test Parameters for Mil-Hdbk-17</b>                  None specified at this time</p>	
<p><b>5. Calculations:</b>                  Compare experimental thermogram to a set of calculated thermograms (see Fig 2 for thermogram example)</p>	

#### 9.4.1.2.2 *Matrix*

There are no national or international standards for thermal diffusivity of CMC matrices.

#### 9.4.1.2.3 *Fiber*

There are no national or international standards for thermal diffusivity of CMC fibers.

#### 9.4.1.2.4 *Interphase*

There are no national or international standards for thermal diffusivity of CMC interphases.

#### 9.4.1.2.5 *Overcoat*

There are no national or international standards for thermal diffusivity of CMC overcoats.

#### 9.4.1.3 *Expansion*

The only national or international standard for thermal expansion of CMCs has been developed for bulk CMCs only.

##### 9.4.1.3.1 *Bulk CMC*

The only national or international standard for thermal expansion of CMCs has been developed by CEN for bulk CMCs only. An overview of CEN ENV 1159-1 "Thermal Expansion of Continuous Fiber Reinforced Ceramic Composites" (see Table 9.4.1.3.1).

**TABLE 9.4.1.3.1** CEN ENV 1159-1 "Thermal Expansion of Continuous Fiber Reinforced Ceramic Composites".

<p><b>1. Outline:</b>  Object Material: continuous fibre reinforced ceramic matrix composite  Test Method: linear measurement  Test Environment: elevated temperature in ambient or inert atmosphere  Number of Test Specimens: per test specimen</p>	<p><b>5. Calculations:</b>  Change in length of test specimen holder, <math>\Delta l</math> (mm) : <math>\Delta l = L_o \bar{\alpha}_a (T_2 - T_1)</math>  where <math>\bar{\alpha}</math> = mean linear thermal expansion coefficient (<math>K^{-1}</math>), <math>L_o</math> = initial length (mm), <math>T_2 - T_1</math> = temperature interval (K), <math>L_o</math> = original test piece length (mm)  Change in length of test specimen, <math>\Delta L</math> (mm): <math>\Delta L = S_{\Delta X} + \Delta l</math> where <math>S_{\Delta X}</math> = measured displacement (mm)  Thermal expansion coefficient (direct), <math>\bar{\alpha}</math> (<math>K^{-1}</math>): <math>\bar{\alpha} = \frac{\Delta l}{L_o(T_2 - T_1)}</math>  where <math>\bar{\alpha}</math> = mean linear thermal expansion coefficient (<math>K^{-1}</math>), <math>L_o</math> = initial length (mm), <math>T_2 - T_1</math> = temperature interval (K)  Thermal expansion coefficient (differential):  <math>\bar{\alpha} = \frac{s\Delta X - \delta}{L_o(T_2 - T_1)} + \bar{\alpha}(R) \frac{L(R)}{L_o}</math> where <math>\delta</math> = baseline correction, <math>L(R)</math> = initial reference piece length (mm), <math>\bar{\alpha}(R)</math> = thermal expansion coefficient for reference piece</p>
<p><b>2. Apparatus:</b>  Test piece holder and push rod shall be made from thermomechanically stable materials of the same type, which shall be chemically inert  Furnace capable of controlled atmosphere  Test mount shall allow free axial movement of test piece and reference piece  Data acquisition system shall measure temperature and displacement to an accuracy of better than 0.1 <math>\mu m</math>  Test piece shall be measured with a device capable of accuracy better than 0.005 mm (micrometer in accordance with annex A or annex B)</p>	<p><b>6. Report:</b>  Name and address of testing establishment  Date of test, unique identification of report  Customer name, address and signatory  Reference to this standard  Description of test material, type of fibre, batch number, date of receipt  Description of test piece; dimensions, preparation method, orientation, mass, volume  Report direct or differential test method  Thermal cycles, number of hold periods, time at hold periods and temperature interval between holds  Maximum temperature testing  hysteresis between heating and cooling cycle  Indication of thermal cycles on the test piece prior to the measurement  Table of the mean linear thermal expansion coefficient at the temperature required  Any features identifiable as phase changes or softening  Comments about test and/or test results</p>
<p><b>3. Procedure:</b>  - Test specimens and reference pieces shall be the same length  - Measure the length of both the test piece and reference piece at room temperature to an accuracy better than 0.2 mm  - Verify calibration of testing machines  - Establish environment if test is to be conducted in an inert environment  - Heating: 1 K/min  - Measure the length of both the test piece and reference piece at elevated temperature to an accuracy better than 0.2 mm  - Cooling rate: 5 K/min</p>	
<p><b>4. Test Parameters for Mil-Hdbk-17</b>  None specified at this time</p>	

#### 9.4.1.3.2 *Matrix*

There are no national or international standards for thermal expansion of CMC matrices.

#### 9.4.1.3.3 *Fiber*

There are no national or international standards for thermal expansion of CMC fibers.

#### 9.4.1.3.4 *Interphase*

There are no national or international standards for thermal expansion of CMC interphases.

#### 9.4.1.3.5 *Overcoat*

There are no national or international standards for thermal expansion of CMC overcoats.

#### 9.4.1.4 *Specific heat*

The only national or international standard for specific heat of CMCs has been developed for bulk CMCs only.

##### 9.4.1.4.1 *Bulk CMC*

The only national or international standard for specific heat of CMCs has been developed by CEN for bulk CMCs only. An overview of CEN ENV 1159-3 "Specific Heat of Continuous Fiber Reinforced Ceramic Composites" (see Table 9.4.1.4.1).

TABLE 9.4.1.4.1 CEN ENV 1159-3 "Specific Heat of Continuous Fiber-Reinforced Ceramic Composites".

<p><b>1. Outline:</b>  Object Material: continuous fibre reinforced ceramic matrix composite  Test Method: drop calorimetry, differential scanning calorimetry  Test Environment: ambient air, at temperature  Number of Test Specimens: minimum of 3 test specimens</p>	<p><b>5. Calculations:</b>  <u>Drop Calorimetry Method:</u>  Calorimeter calibration factor, K: <math>K = \frac{\text{power dissipated}}{\text{calorimetric output signal}}</math> where  Q = Heat variation: <math>Q_i = K S_i</math> where <math>S_i</math> = calorimetric output signal  Mean specific heat capacity, cp: <math>\bar{C}_p(T_2 - T_1) = \frac{1}{m_i} \frac{Q_i(T_1 &gt; T_2)}{(T_2 - T_1)}</math> where  <math>m_i</math> = mass (test or reference piece, or crucible) (g), <math>Q_i</math> = heat variation,  <math>T_2</math> = calorimeter temperature (K), <math>T_1</math> = test piece initial temperature (K)  <u>Differential method:</u>  Heat quantity: <math>Q_E = \int_0^t P_E dt = (m_t \bar{C}_p(T_1, T_2) + C_C - C_O)(T_2 - T_1)</math>  Heat capacity: <math>Q_B = \int_0^t P_B dt = [C_C + C_O](T_2 - T_1)</math>  Where <math>P_E</math>, <math>P_B</math> = thermal power for test and empty crucible respectively, <math>m_t</math> = mass of test piece (g), <math>C_o</math> = heat capacity of calorimeter, <math>C_c</math> = heat capacity of crucible  Mean specific heat capacity: <math>\bar{C}_p(T_1, T_2) = \frac{Q_E - Q_B}{m_t(T_2 - T_1)}</math>  <u>Continuous method:</u>  Heat quantity: <math>P_E = (m_t C_p + C_C + C_O)\beta</math>  Heat capacity: <math>P_B = (C_C + C_O)\beta</math>  Mean specific heat capacity: <math>C_p = \frac{P_E - P_B}{m_t \beta}</math>  <u>Differential Scanning Calorimetry Method:</u>  <math>KS_C = (C_C + C_O)\beta</math>  <math>KS_{C+t} = (m_t C_{p_t} + C_C + C_O)\beta</math>  <math>KS_{C+r} = (m_r C_{p_r} + C_C + C_O)\beta</math>  Mean specific heat capacity: <math>C_{p_t} = C_{p_r} \frac{m_r S_{C+t} - S_C}{m_t S_{C+r} - S_C}</math>  K = calorimeter calibration factor, <math>C_o</math> = heat capacity of calorimeter,  <math>C_c</math> = heat capacity of crucible, <math>C_{p_t}</math> = mean specific heat capacity of test piece, <math>C_{p_r}</math> = mean specific heat capacity of reference piece, <math>m_r</math> = mass of reference piece, <math>m_t</math> = mass of test piece, S = output signal with respect to crucible, test piece and reference piece (mV)</p>
<p><b>2. Apparatus:</b>  Drop calorimeter, balance with accuracy of 0.1 mg, thermocouples in accordance to HD 446.1, data acquisition system  Differential scanning calorimeter, balance with accuracy of 0.1 mg, thermocouples in accordance to HD 446.1, data acquisition system</p>	
<p><b>3. Procedure:</b>  <u>Drop Calorimetry Method:</u>  - Calibrate equipment  - Dry test specimen and reference piece at 373 +/- 5 K  - Measure mass when a crucible is not used to +/- 0.1 mg  - Measure mass of each assembly dropped when crucible is used (empty crucible, crucible and reference piece, crucible and test piece)  - Place test piece and in condition chamber at temperature and wait 15 min for test/reference piece to reach thermal equilibrium  - Measure <math>T_1</math> and <math>T_2</math> temperatures  - Repeat for reference piece  <u>Differential Scanning Calorimetry Method:</u>  - Baseline procedure;  - Weigh two empty crucibles  - Place two crucibles in chamber  - Set calorimeter heating rate ( between 1 K/min and 20 K/min), initial and final temperature  - Heat to initial temperature, wait for stabilization, record initial temperature  - Heat to final temperature  - Record base line final temperature  - Cool to initial temperature  - Weigh test piece and place in crucible  - Repeat baseline procedure with test piece in test chamber  - Repeat baseline procedure with reference piece in test chamber</p>	
<p><b>4. Test Parameters for Mil-Hdbk-17</b>  None specified at this time</p>	

**TABLE 9.4.1.4.1** CEN ENV 1159-3 "Specific Heat of Continuous Fiber-Reinforced Ceramic Composites" (cont'd).

<p><b>6. Report:</b></p> <p>Name and address of testing establishment Date of test, unique identification of report Customer name, address and signatory Reference to this standard Brief details of equipment used Calibration procedures Description of test material, type of fibre, batch number, date of receipt, method of manufacture Relevant test parameters Results of individual tests Mean results and standard deviation (if required) Comments about test and/or test results Value of mean specific heat capacity between two temperatures calculated on at least three test specimens</p>	
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*9.4.1.4.2 Matrix*

There are no national or international standards for specific heat of CMC matrices.

*9.4.1.4.3 Fiber*

There are no national or international standards for specific heat of CMC fibers.

*9.4.1.4.4 Interphase*

There are no national or international standards for specific heat of CMC interphases.

*9.4.1.4.5 Overcoat*

There are no national or international standards for specific heat of CMC overcoats.

*9.4.1.5 Thermal shock*

There are no national or international standards for thermal shock of bulk CMCs or their constituents.

*9.4.1.5.1 Bulk CMC*

There are no national or international standards for thermal shock of bulk CMCs.

*9.4.1.5.2 Matrix*

There are no national or international standards for thermal shock of CMC matrices.

*9.4.1.5.3 Fiber*

There are no national or international standards for thermal shock of CMC fibers.

*9.4.1.5.4 Interphase*

There are no national or international standards for thermal shock of CMC interphases.

*9.4.1.5.5 Overcoat*

There are no national or international standards for thermal shock of CMC overcoats.

*9.4.1.6 Thermal fatigue*

There are no national or international standards for thermal fatigue of bulk CMCs or their constituents.

*9.4.1.6.1 Bulk CMC*

There are no national or international standards for thermal fatigue of bulk CMCs.

*9.4.1.6.2 Matrix*

There are no national or international standards for thermal fatigue of CMC matrices.

*9.4.1.6.3 Fiber*

There are no national or international standards for thermal fatigue of CMC fibers.

#### 9.4.1.6.4 *Interphase*

There are no national or international standards for thermal fatigue of CMC interphases.

#### 9.4.1.6.5 *Overcoat*

There are no national or international standards for thermal fatigue of CMC overcoats.

### 9.4.2 **Mechanical**

Mechanical test methods include those which relate to mechanical properties which directly influence a material's design use. In particular, tension, compressive, shear, flexure, and fracture of flat plates are highlighted.

#### 9.4.2.1 *Tension*

Numerous tensile test methods exist for CMCs and their constituents. These include those for in-plane and trans-thickness, monotonic strength (ambient and elevated temperatures), cyclic fatigue (ambient and elevated temperatures), and creep. The following sections contain specifics about test methods required to qualify CMC materials for inclusion in MIL-HDBK-17.

##### 9.4.2.1.1 *Bulk CMC*

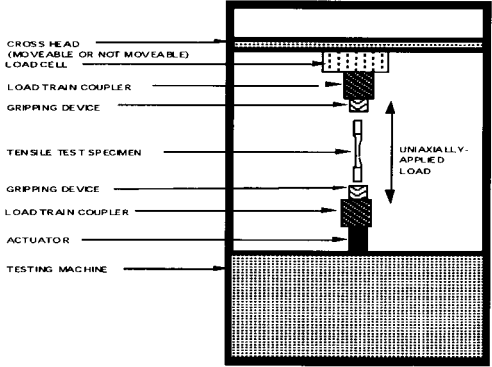
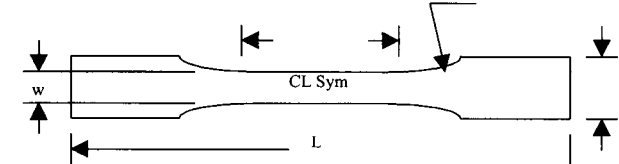
The following list of test methods and corresponding tables contain specific test parameters to be used for tensile testing bulk CMCs.

##### 9.4.2.1.1.1 *In-plane monotonic tensile strength (ambient temperature)*

In-plane monotonic tensile strength (ambient temperature): ASTM C 1275-95 "Standard Test Method for Monotonic Tensile Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross Sections at Ambient Temperatures" (see Table 9.4.2.1.1.1).



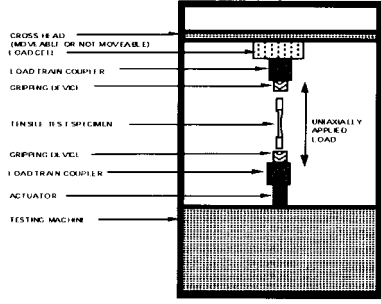
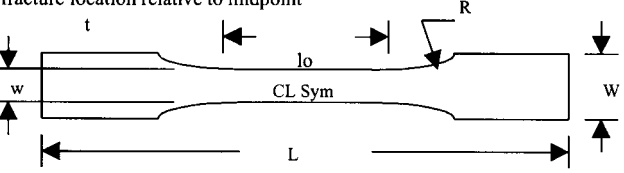
**TABLE 9.4.2.1.1.1** ASTM C1275-95 “Standard Test Method for Monotonic Tensile Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross Sections at Ambient Temperatures”.

<p><b>1. Outline:</b>                  Object Material: continuous fibre reinforced ceramic composite                  Test Method: in-plane monotonic tensile strength                  Test Environment: ambient temperature, ambient air                  Number of Test Specimens: minimum of 5 valid tests</p>	<p><b>4. Test Parameters for Mil-Hdbk-17</b>                  None specified at this time</p>
<p><b>2. Apparatus:</b>                  Testing machine shall conform to the requirements of ASTM Practice E 4                  Extensometry shall conform to the requirements of ASTM Practice E83</p> 	<p><b>5. Calculations:</b>                  Engineering stress, <math>\sigma</math> (MPa): <math>\sigma = \frac{P}{A}</math> where P = applied, force (N), A = original cross sectional area (<math>\text{mm}^2</math>) where <math>A = wt</math> (<math>\text{mm}^2</math>)                  Engineering strain, <math>\epsilon</math> (m/m): <math>\epsilon = \frac{(l-l_0)}{l_0}</math> where <math>\epsilon</math> = engineering strain, l = gage length at any time (mm), <math>l_0</math> = original gage length (mm)                  Tensile strength, <math>S_u</math> (MPa): <math>S_u = \frac{P_{\max}}{A}</math> where <math>P_{\max}</math> = maximum load (N)                  Strain at tensile strength, <math>\epsilon_u</math> (m/m): <math>\epsilon_u = \epsilon</math> at <math>S_u</math>                  Fracture strength, <math>S_f</math> (MPa): <math>S_f = \frac{P_{\text{fracture}}}{A}</math> where <math>P_{\text{fracture}}</math> = fracture load (N)                  Strain at fracture strength, <math>\epsilon_f</math> (m/m): <math>\epsilon_f = \epsilon</math> at <math>S_f</math>                  Elastic modulus, E (MPa): <math>E = \frac{\Delta\sigma}{\Delta\epsilon}</math> where <math>\Delta\sigma/\Delta\epsilon</math> = the linear slope of the <math>\sigma - \epsilon</math> curve                  Poisson's ratio, <math>\nu</math>: <math>\nu = \frac{\Delta\epsilon_T}{\Delta\epsilon_L}</math> where <math>\Delta\epsilon_T/\Delta\epsilon_L</math> = slope of the linear region of plot of transverse strain vs. longitudinal strain                  Modulus of resilience, <math>U_R</math> (MJ/m<sup>3</sup>): <math>U_R = \int_0^{\epsilon_f} \sigma d\epsilon \approx \frac{1}{2} \sigma_o \epsilon_o</math>                  Modulus of toughness, <math>U_T</math> (MJ/m<sup>3</sup>): <math>U_T = \int_0^{\epsilon_f} \sigma d\epsilon \approx \frac{\sigma_o + S_u}{2}</math></p> <p>Mean: <math>\bar{X} = \frac{\sum_{i=1}^n x_i}{n}</math> Standard deviation: <math>s.d. = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{X})^2}{n-1}}</math></p>
<p><b>3. Procedure:</b>                  Test specimen dimensions (see Fig. 2): measure thickness &amp; width on at least three different cross sectional planes in the gage section                  Test rate: 0.1 mm/min or sufficient to produce final fracture in 5 to 10 s                  Procedure: record load vs. displacement or stress vs. strain; measure and report fracture location relative to midpoint</p>  <p>Fig. 2 Example of a test specimen geometry</p>	<p><b>6. Report:</b>                  Test Set:                  - date and location of testing                  - number of test specimens and geometry                  - type, configuration: test machine, extensometry, grip, load train                  - conditions: temperature, relative humidity, atmosphere, test mode                  - mean, standard deviation, coefficient of variation for: tensile strength, strain at tensile strength, fracture strength, strain at fracture strength, elastic modulus                  Individual Test Specimen:                  - overall test specimen dimensions, average cross-sectional dimensions, average surface roughness                  - tensile strength, strain at tensile strength, fracture strength, strain at fracture strength, elastic modulus, plot of stress-strain curve, elastic modulus</p>

*9.4.2.1.1.2 In-plane monotonic tensile strength (elevated temperature)*

In-plane monotonic tensile strength (elevated temperature): ASTM C 1359-97 "Standard Test Method for Monotonic Tensile Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross Sections at Elevated Temperatures" (see Table 9.4.2.1.1.2).

**TABLE 9.4.2.1.1.2** ASTM C1359-97 "Standard Test Method for Monotonic Tensile Strength Testing of Continuous Fiber Reinforced Advanced Ceramics with Solid Rectangular Cross Sections at Elevated Temperatures".

<p><b>1. Outline:</b>                  Object Material: continuous fibre reinforced ceramic composite                  Test Method: in-plane monotonic tensile strength                  Test Environment: elevated temperature, ambient air                  Number of Test Specimens: minimum of 5 valid tests</p>	<p><b>4. Test Parameters for Mil-Hdbk-17</b>                  None specified at this time</p>
<p><b>2. Apparatus:</b>                  Testing machine shall conform to the requirements of ASTM Practice E 4                  Extensometry shall conform to the requirements of ASTM Practice E83</p>  <p>Fig. 1 Schematic illustration of tensile testing machine</p>	<p><b>5. Calculations:</b>                  Engineering stress, <math>\sigma</math> (MPa): <math>\sigma = \frac{P}{A}</math> where P = applied, force (N), A = original cross sectional area (<math>\text{mm}^2</math>) where <math>A = wt</math> (<math>\text{mm}^2</math>)                  Engineering strain, <math>\epsilon</math> (m/m): <math>\epsilon = \frac{(l-l_0)}{l_0}</math> where <math>\epsilon</math> = engineering strain, l = gage length at any time (mm), <math>l_0</math> = original gage length (mm)                  Tensile strength, <math>S_u</math> (MPa): <math>S_u = \frac{P_{max}}{A}</math> where <math>P_{max}</math> = maximum load (N)                  Strain at tensile strength, <math>\epsilon_u</math> (m/m): <math>\epsilon_u = \epsilon</math> at <math>S_u</math>                  Fracture strength, <math>S_f</math> (MPa): <math>S_f = \frac{P_{fracture}}{A}</math> where <math>P_{fracture}</math> = fracture load (N)                  Strain at fracture strength, <math>\epsilon_f</math> (m/m): <math>\epsilon_f = \epsilon</math> at <math>S_f</math>                  Elastic modulus, E (MPa): <math>E = \frac{\Delta\sigma}{\Delta\epsilon}</math> where <math>\Delta\sigma/\Delta\epsilon</math> = the linear slope of the <math>\sigma - \epsilon</math> curve                  Poisson's ratio, <math>\nu</math>: <math>\nu = \frac{\Delta\epsilon_T}{\Delta\epsilon_L}</math> where <math>\Delta\epsilon_T/\Delta\epsilon_L</math> = slope of the linear region of plot of transverse strain vs. longitudinal strain                  Modulus of resilience, <math>U_R</math> (<math>\text{MJ/m}^3</math>): <math>U_R = \int_0^{\epsilon_f} \sigma d\epsilon \approx \frac{1}{2} \sigma_o \epsilon_o</math>                  Modulus of toughness, <math>U_T</math> (<math>\text{MJ/m}^3</math>): <math>U_T = \int_0^{\epsilon_f} \sigma d\epsilon \approx \frac{\sigma_o + S_u}{2}</math>                  Mean: <math>\bar{X} = \frac{\sum_{i=1}^n x_i}{n}</math> Standard deviation: <math>s.d. = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{X})^2}{n-1}}</math></p>
<p><b>3. Procedure:</b>                  Test Specimen Dimensions (see Fig. 2): measure thickness &amp; width on at least three different cross sectional planes in the gage section                  Test Rate: 0.1 mm/min or sufficient to produce final fracture in 5 to 10 s                  Temperature limits: indicated <math>\leq 1273 \pm 3</math> K; nominal <math>&gt; 1273 \pm 6</math> K                  gage section <math>\leq 773 \pm 5</math> K; <math>&gt; 773 \pm 1\%</math> of test temp                  Temperature rate and hold; <math>&lt; 50</math> K/min, hold time <math>\leq 30</math> min                  Procedure: record load vs. displacement or stress vs. strain; measure and report fracture location relative to midpoint</p>  <p>Fig 2 Example of test specimen geometry</p>	<p><b>6. Report:</b>                  Test Set:                  - date and location of testing                  - number of test specimens and geometry                  - type, configuration: test machine, extensometry, grip, load train                  - conditions: temperature, relative humidity, atmosphere, test mode                  - mean, standard deviation, coefficient of variation for: tensile strength, strain at tensile strength, fracture strength, strain at fracture strength, elastic modulus                  Individual Test Specimen:                  - overall test specimen dimensions, average cross-sectional dimensions, average surface roughness                  - tensile strength, strain at tensile strength, fracture strength, strain at fracture strength, elastic modulus, plot of stress-strain curve, elastic modulus</p>

*9.4.2.1.1.3 Trans-thickness monotonic tensile strength (ambient temperature)*

Trans-thickness monotonic tensile strength (ambient temperature): no standard currently exists.

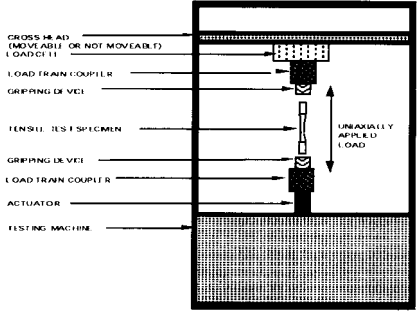
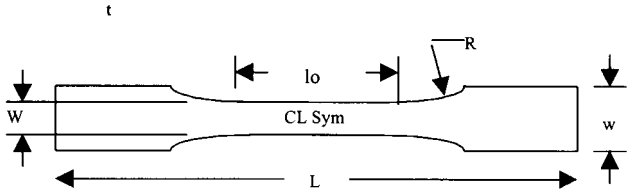
*9.4.2.1.1.4 Trans-thickness monotonic tensile strength (elevated temperature)*

Trans-thickness monotonic tensile strength (elevated temperature): no standard currently exists.

*9.4.2.1.1.5 Cyclic fatigue (ambient temperature)*

Cyclic fatigue (ambient temperature): ASTM C 1360-97 "Standard Practice for Constant-Amplitude, Axial, Tension-Tension Cyclic Fatigue of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperatures" (see Table 9.4.2.1.1.5).

**TABLE 9.4.2.1.1.5** ASTM C1360-97 "Standard Practice for Constant-Amplitude, Axial, Tension-Tension Cyclic Fatigue of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperature".

<p><b>1. Outline:</b>                  Object Material: continuous fibre reinforced ceramic matrix composite                  Test Method: cyclic fatigue                  Test Environment: ambient temperature, ambient air                  Number of Test Specimens: reference STP 91-A</p>	<p><b>4. Test Parameters for Mil-Hdbk-17</b>                  None specified at this time</p>
<p><b>2. Apparatus:</b>                  Testing machine shall conform to the requirements of ASTM Practice E 4                  Extensometry shall conform to the requirements of ASTM Practice E83</p>  <p>Fig. 1 Schematic illustration of tensile testing machine</p>	<p><b>5. Calculations:</b>                  Engineering stress, <math>\sigma</math> (MPa): <math>\sigma = \frac{P}{A}</math> where P = applied, force (N), A = original cross sectional area (<math>\text{mm}^2</math>) where <math>A = wt</math> (<math>\text{mm}^2</math>)                  Engineering strain, <math>\epsilon</math> (m/m): <math>\epsilon = \frac{l - l_0}{l_0}</math> where <math>\epsilon</math> = engineering strain, <math>l</math> = gage length at any time (mm), <math>l_0</math> = original gage length (mm)                  Maximum stress, <math>\sigma_{\max}</math> (MPa): <math>\sigma_{\max} = \frac{P_{\max}}{A}</math> where <math>P_{\max}</math> = maximum applied cyclic load (N)                  Minimum stress, <math>\sigma_{\min}</math> (MPa): <math>\sigma_{\min} = \frac{P_{\min}}{A}</math> where <math>P_{\min}</math> = minimum applied cyclic load (N)                  Stress ratio, R: <math>R = \frac{\sigma_{\min}}{\sigma_{\max}}</math>                  Elastic modulus, E (MPa): <math>E = \frac{\Delta\sigma}{\Delta\epsilon}</math> where <math>\Delta\sigma/\Delta\epsilon</math> = the slope of the <math>\sigma - \epsilon</math> curve in the linear region                  Mean: <math>\bar{X} = \frac{\sum X_i}{n}</math>, Standard deviation: <math>s.d. = \sqrt{\frac{\sum (X_i - \bar{X})^2}{n-1}}</math></p>
<p><b>3. Procedure:</b>                  Test Specimen Dimensions (See Fig. 2): measure thickness &amp; width on at least three different cross sectional planes in the gage section                  Test Rate: sine wave, R = 0.1, frequency = 1-10 Hz.                  Maximum Stresses: Percentages of average tensile strength from ASTM Test Method C1275                  Procedure: Preload test specimen; record preload, number of cycles, fracture location</p>  <p>Fig 2 Example of a test specimen geometry</p>	<p><b>6. Report:</b>                  Test Set:</p> <ul style="list-style-type: none"> <li>• date and location of testing</li> <li>• number of test specimens and geometry</li> <li>• type, configuration: test machine, strain measurement equipment, grip interface, load train couplers, heating system, temperature measuring system</li> <li>• conditions: temperature, relative humidity, atmosphere, test mode</li> <li>• mean, standard deviation, coefficient of variation for following properties: strain-life, stress-life, tensile strength, strain at tensile strength, fracture strength, strain at fracture strength, elastic modulus</li> </ul> <p>Individual Test Specimens:</p> <ul style="list-style-type: none"> <li>• overall test specimen dimensions, average cross-sectional dimensions, average surface roughness</li> <li>• strain-life, stress-life, R ratio max/min cyclic stress, frequency number of cycles, amplitude stress, wave form</li> </ul>

*9.4.2.1.1.6 Cyclic fatigue (elevated temperature)*

Cyclic fatigue (elevated temperature): no standard currently exists.

*9.4.2.1.1.7 Creep*

Creep: ASTM C 1337-96 "Standard Test Method for Creep and Creep Rupture of Continuous Fiber-Reinforced Advanced Ceramics under Tensile Loading" (see Table 9.4.2.1.1.7).



#### 9.4.2.1.2 *Matrix*

There are no national or international standards for tensile testing of CMC matrices.

#### 9.4.2.1.3 *Fiber*

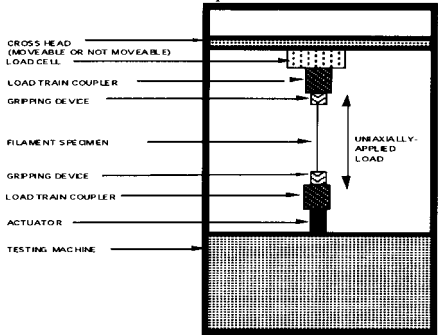
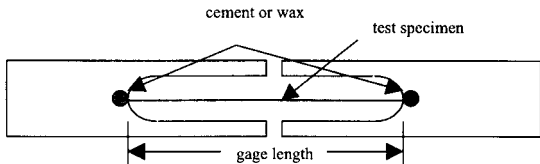
The following list of test methods and corresponding tables contain specific test parameters to be used for tensile testing CMC fibers.

##### 9.4.2.1.3.1 *Monotonic tensile strength (ambient temperature)*

Monotonic tensile strength (ambient temperature): ASTM D 3379-75 "Standard Test Method For Tensile Strength and Young's Modulus for High-Modulus Single-Filament Materials" (see Table 9.4.2.1.3.1).



**TABLE 9.4.2.1.3.1** ASTM D 3379-75 "Standard Test Method for Tensile Strength and Young's Modulus for High-Modulus Single-Filament Materials.

<p><b>1. Outline:</b>                  Object Material: fibres for continuous fibre reinforced ceramic matrix composite                  Test Method: tensile strength                  Test Environment: ambient temperature, ambient air                  Number of Test Specimens: minimum of 10</p> <p><b>2. Apparatus:</b>                  Testing Machine shall conform to the requirements of ASTM Practice E 4</p>  <p style="text-align: center;">Fig. 1 Schematic illustration of tensile testing machine</p>	<p><b>4. Test Parameters for Mil-Hdbk-17</b>                  None specified at this time</p> <p><b>5. Calculations:</b>                  Area, A (m<sup>2</sup>): <math>A = \frac{\sum a_f \times 10^{-6}}{(N)(M_f)^2}</math> where A = average fibre area (m<sup>2</sup>), a<sub>f</sub> = area of one fibre (m<sup>2</sup>), N = number of fibres, M<sub>f</sub> = photomicrograph magnification factor</p> <p>Tensile strength, T (Pa): T = F/A where F = failure load (N), A = average fibre area (m<sup>2</sup>)</p> <p>Indicated compliance, C<sub>a</sub> (mm/N): C<sub>a</sub> = l/P x H/S where l = total extension (mm), P = full scale force (N), H = crosshead speed (mm/s), S = chart speed (mm/s)</p> <p>True compliance, C (mm/N): C = C<sub>a</sub> - C<sub>s</sub> where C<sub>s</sub> = system compliance (mm/N)</p> <p>Elastic (Young's) modulus Y<sub>m</sub>=E (Pa): Y<sub>m</sub> = L/CA where L = test specimen gage length (mm)</p> <p>Mean: <math>\bar{X} = \frac{\sum_{i=1}^n X_i}{n}</math>, Standard deviation: s.d. = <math>\sqrt{\frac{\sum_{i=1}^n (X_i - \bar{X})^2}{n-1}}</math></p>
<p><b>3. Procedure:</b>                  Test specimen dimensions: determine average dimensions are for test group from photomicrographs                  Calibration of test machine: Initial plus every four hours during testing                  Test rate: crosshead speed sufficient to give failure within 1 min                  Test specimen mounting: grip one side, cut/burn tab, grip other side                  Testing: break test specimen, record failure load and elongation</p>  <p style="text-align: center;">Fig. 2 Example of test specimen mounting</p>	<p><b>6. Report:</b></p> <ul style="list-style-type: none"> <li>• identification of test specimens: material type/source, manufacturer name and code number, previous material history, etc.</li> <li>• method of selecting test specimens, number of test specimens, method of mounting test specimens, method of measuring cross-sectional area</li> <li>• cross-sectional areas, test specimen gage length, system compliance</li> <li>• crosshead speed, chart speed, load cell, etc.</li> <li>• method for determining gage length, breaking loads</li> <li>• individual results, average, standard deviation for following properties:</li> <li>• Young's modulus, tensile strength</li> <li>• environmental conditions: temperatre, relative humidity, atmosphere</li> <li>• date of test</li> </ul>

#### 9.4.2.1.3.2 *Monotonic tensile strength (elevated temperature)*

Monotonic tensile strength (elevated temperature): no standard currently exists.

#### 9.4.2.1.4 *Interphase*

There are no national or international standards for tensile testing of CMC interphases.

#### 9.4.2.1.5 *Overcoat*

There are no national or international standards for tensile testing of CMC overcoats.

### 9.4.2.2 *Compression*

Few compressive test methods exist for CMCs and their constituents. These include in-plane monotonic strength (ambient and elevated temperatures). Eventually they will include in-plane and trans-thickness, monotonic strength (ambient and elevated temperatures), cyclic fatigue (ambient and elevated temperatures), and creep. The following sections contain specifics about test methods required to qualify materials for inclusion in MIL-HDBK-17.

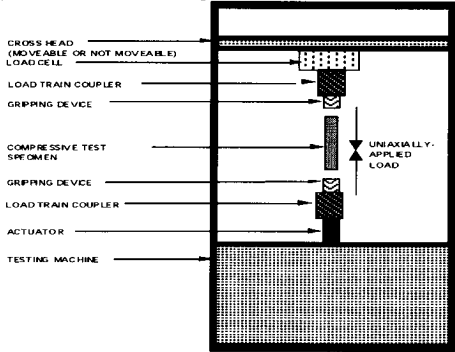
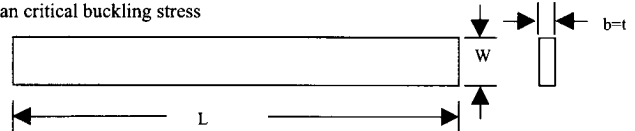
#### 9.4.2.2.1 *Bulk CMC*

The following list of test methods and corresponding tables contain specific test parameters to be used for compression testing bulk CMCs.

#### 9.4.2.2.2 *In-plane monotonic compressive strength (ambient temperature)*

In-plane monotonic compressive strength (ambient temperature): ASTM C 1258-97 "Standard Test Method for Monotonic Compressive Strength Testing of Continuous Fiber-Reinforced Advanced Ceramics with Solid Rectangular Cross Sections at Ambient Temperatures" (see Table 9.4.2.2.2).

**TABLE 9.4.2.2.2** *ASTM C1258-97 “Standard Test Method for Monotonic Compressive Strength Testing of Continuous Fiber Reinforced Advanced Ceramics with Solid Rectangular Cross Sections at Ambient Temperatures”.*

<p><b>1. Outline:</b>                  Object Material: continuous fibre reinforced ceramic matrix composite                  Test Method: in-plane monotonic compressive strength                  Test Environment: ambient temperature, ambient air                  Number of Test Specimens: minimum of 5 valid tests</p> <p><b>2. Apparatus:</b>                  Testing machine shall conform to the requirements of ASTM Practice E 4                  Extensometry shall conform to the requirements of ASTM Practice E83</p>  <p style="text-align: center;">Fig. 1 Schematic illustration of a testing machine</p>	<p><b>4. Test Parameters for Mil-Hdbk-17</b>                  None specified at this time</p> <p><b>5. Calculations:</b>                  Slenderness ratio: <math>\frac{l}{k} = \sqrt{12} \frac{l}{b}</math> where <math>l</math> = length of gage section, <math>k</math> = least radius of gyration for the cross section, <math>b</math> = thickness of the cross section</p> <p>Critical buckling stress, <math>\sigma_{cr}</math> (MPa): <math>\sigma_{cr} = \frac{P_{cr}}{wb} = \frac{4\pi^2 EI}{l^2 wb}</math> <math>P_{cr}</math> = critical compressive load (N), <math>w</math> = test specimen width, <math>t</math> = test specimen thickness, <math>E</math> = longitudinal elastic modulus, <math>I</math> = moment of inertia in <math>b</math> direction where <math>wb^3/12</math>, <math>l</math> = free (unsupported) length of test specimen gage section</p> <p>Engineering stress, <math>\sigma</math> (MPa): <math>\sigma = \frac{P}{A}</math> where <math>P</math> = applied, uniaxial load (N), <math>A</math> = cross-sectional area (<math>\text{mm}^2</math>) where <math>A = wt</math> (<math>\text{mm}^2</math>)</p> <p>Engineering strain, <math>\epsilon</math> (m/m): <math>\epsilon = \frac{(l - l_0)}{l_0}</math> where <math>l</math> = gage length at any time (mm), <math>l_0</math> = original gage length (mm)</p> <p>Compressive strength, <math>S_u</math> (MPa): <math>S_u = \frac{P_{max}}{A}</math> where <math>P_{max}</math> = breaking load (N)</p> <p>Elastic modulus, <math>E</math> (MPa): <math>E = \frac{\Delta\sigma}{\Delta\epsilon}</math> where <math>\Delta\sigma/\Delta\epsilon</math> = the slope of the <math>\sigma - \epsilon</math> curve in the linear region</p> <p>Mean: <math>\bar{X} = \frac{\sum_{i=1}^n x_i}{n}</math>, Standard deviation: <math>s.d. = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{X})^2}{n-1}}</math></p>
<p><b>3. Procedure:</b>                  Test Specimen Dimensions (see Fig. 2): slenderness ratio, <math>l/k \leq 30</math>; measure thickness &amp; width on at least three different cross sectional planes in the gage section                  Test rate: sufficient to cause final fracture in 5 to 10 s                  Test mode: displacement or load control in a single ramp                  Testing: record stress, strain, fracture location, temperature, relative humidity, check to make sure that fracture occurred within gage section, fracture stress less than critical buckling stress</p>  <p style="text-align: center;">Fig. 2 Example of a test specimen geometry</p>	<p><b>6. Report:</b>                  Test Set:</p> <ul style="list-style-type: none"> <li>• date and location of testing</li> <li>• number of test specimens and geometry</li> <li>• type, configuration: test machine, strain measurement equipment, grip interface, load train couplers</li> <li>• conditions: temperature, relative humidity, atmosphere, test mode</li> <li>• mean, standard deviation, coefficient of variation for following properties: tensile strength, strain at tensile strength, fracture strength, strain at fracture strength, elastic modulus</li> </ul> <p>Individual Test Specimens:</p> <ul style="list-style-type: none"> <li>• overall test specimen dimensions, average cross-sectional dimensions, average surface roughness</li> <li>• compressive strength, strain at compressive strength, fracture strength, strain at fracture strength, elastic modulus, plot of stress-strain curve</li> </ul>

#### 9.4.2.2.3 *Matrix*

There are no national or international standards for compressive testing of CMC matrices.

#### 9.4.2.2.4 *Fiber*

There are no national or international standards for compressive testing of CMC fibers.

#### 9.4.2.2.5 *Interphase*

There are no national or international standards for compressive testing of CMC interphases.

#### 9.4.2.2.6 *Overcoat*

There are no national or international standards for compressive testing of CMC overcoats.

#### 9.4.2.3 *Shear*

Several shear test methods exist for CMCs and their constituents. These include in-plane and interlaminar shear strength (ambient temperatures). Eventually they will include in-plane and interlaminar shear strength at elevated temperatures as well as interfacial shear strength at both ambient and elevated temperatures. The following sections contain specifics about test methods required to qualify materials for inclusion in MIL-HDBK-17.

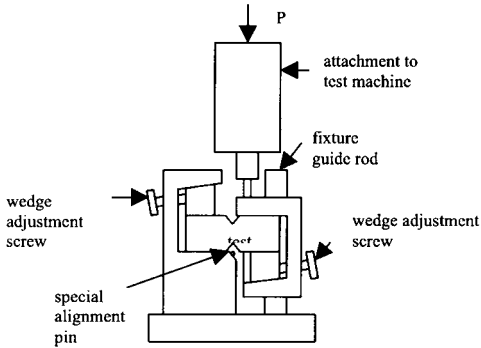
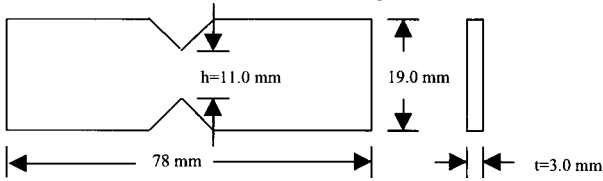
##### 9.4.2.3.1 *Bulk CMC*

The following list of test methods and corresponding tables contain specific test parameters to be used for shear testing bulk CMCs.

##### 9.4.2.3.1.1 *In-plane monotonic shear strength (ambient temperature)*

In-plane monotonic shear strength (ambient temperature): ASTM C 1292-95 "Standard Test Method for Shear Strength of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperatures" (see Table 9.4.2.3.1.1).

**TABLE 9.4.2.3.1.1** ASTM C1292-95 "Standard Test Method for In-Plane (Iosipescu) Shear Strength of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperatures".

<p><b>1. Outline:</b>                  Object Material: continuous fibre reinforced ceramic matrix composite                  Test Method: in-plane shear strength                  Test Environment: ambient temperature, ambient air                  Number of Test Specimens: minimum of 10 valid tests</p>	<p><b>4. Test Parameters for Mil-Hdbk-17</b>                  None specified at this time</p>
<p><b>2. Apparatus:</b>                  Testing Machine shall conform to the requirements of ASTM Practice E 4</p>  <p>Fig. 1 Schematic illustration of in-plane shear test fixture</p>	<p><b>5. Calculations:</b>                  In-plane shear strength, <math>\tau</math> (MPa): <math>\tau = \frac{P_{max}}{A}</math> where <math>A = t \cdot h =</math> shear stressed area (<math>mm^2</math>), <math>h =</math> distance between the notches (mm), <math>t =</math> average test specimen thickness (mm)</p> <p>Mean: <math>\bar{X} = \frac{\sum X_i}{n}</math>, Standard deviation: <math>s.d. = \sqrt{\frac{\sum (X_i - \bar{X})^2}{n-1}}</math></p>
<p><b>3. Procedure:</b>                  Test specimen dimensions (see Fig. 2): measure thickness and width on at least three different cross sectional planes in the gage section                  Crosshead speed: 0.05mm/min or sufficient to produce final fracture in 10-30 s                  Record load vs. displacement or stress vs. strain                  Measure and report fracture location relative to midpoint</p>  <p>Fig. 2 Test specimen geometry</p>	<p><b>6. Report:</b>                  Test Set:</p> <ul style="list-style-type: none"> <li>• date and location of testing</li> <li>• number of test specimens and geometry</li> <li>• type, configuration: test machine, strain measurement equipment, test fixture</li> <li>• conditions: temperature, relative humidity, atmosphere, test mode</li> <li>• mean, standard deviation, coefficient of variation for following properties: shear strength, strain at shear strength</li> </ul> <p>Individual Test Specimen:</p> <ul style="list-style-type: none"> <li>• overall test specimen dimensions, average cross-sectional dimensions, average surface roughness</li> <li>• shear strength, strain at shear strength, plot of stress-strain curve</li> </ul>

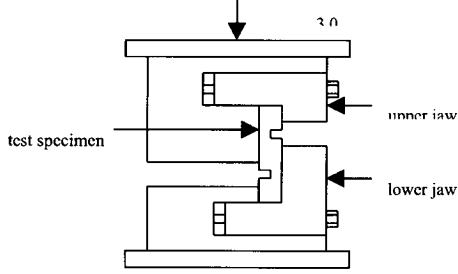
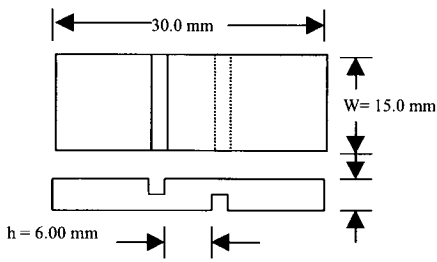
*9.4.2.3.1.2 In-plane monotonic shear strength (elevated temperature)*

In-plane monotonic shear strength (elevated temperature): no standard exists.

*9.4.2.3.1.3 Interlaminar monotonic shear strength (ambient temperature)*

Interlaminar monotonic shear strength (ambient temperature): ASTM C 1292-95 "Standard Test Method for Shear Strength of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperatures" (see Table 9.4.2.3.1.3).

**TABLE 9.4.2.3.1.3** ASTM C1292-95 “Standard Test Method for Interlaminar (Double-Notch Compression) Shear Strength of Continuous Fiber-Reinforced Advanced Ceramics at Ambient Temperatures”.

<p><b>1. Outline:</b>                  Object Material: continuous fibre reinforced ceramic matrix composite                  Test Method: interlaminar shear strength                  Test Environment: ambient temperature, ambient air                  Number of Test Specimens: minimum of 10 valid tests</p>	<p><b>4. Test Parameters for Mil-Hdbk-17</b>                  None specified at this time</p>
<p><b>2. Apparatus:</b>                  Testing Machine shall conform to the requirements of ASTM Practice E 4</p>  <p>Fig. 1 Schematic illustration of an interlaminar shear strength test fixture</p>	<p><b>5. Calculations:</b>                  Interlaminar shear strength, <math>\tau</math> (MPa): <math>\tau = \frac{P_{max}}{A}</math> where <math>A = W h</math> = shear stressed area (<math>mm^2</math>), <math>W</math> = average test specimen width (mm), <math>h</math> = distance between the notches (mm)</p> <p>Mean: <math>\bar{X} = \frac{\sum_{i=1}^n X_i}{n}</math>, Standard deviation: <math>s.d. = \sqrt{\frac{\sum_{i=1}^n (X_i - \bar{X})^2}{n-1}}</math></p>
<p><b>3. Procedure:</b>                  Test specimen dimensions (see Fig. 2): measure thickness &amp; width on at least three different cross sectional planes in the gage section                  Crosshead speed: 0.05 mm/min or sufficient to produce final fracture in 10 to 30 s                  Record load vs. displacement or stress vs. strain                  Measure and report fracture location relative to midpoint</p>  <p>Fig. 2 Test specimen geometry</p>	<p><b>6. Report:</b>                  Test Set:</p> <ul style="list-style-type: none"> <li>• date and location of testing</li> <li>• number of test specimens and geometry</li> <li>• type, configuration: test machine, strain (displacement) measurement device, test fixture</li> <li>• conditions: temperature, relative humidity, atmosphere, test mode</li> <li>• mean, standard deviation, coefficient of variation for following properties: shear strength, strain at shear strength</li> </ul> <p>Individual Test Specimen:</p> <ul style="list-style-type: none"> <li>• overall test specimen dimensions, average cross-sectional dimensions, average surface roughness</li> <li>• shear strength, strain at shear strength, plot of stress-strain curve</li> </ul>

#### 9.4.2.3.1.4 *Interlaminar monotonic shear strength (elevated temperature)*

Interlaminar monotonic shear strength (elevated temperature): no standard exists.

#### 9.4.2.3.2 *Matrix*

There are no national or international standards for shear testing of CMC matrices.

#### 9.4.2.3.3 *Fiber*

There are no national or international standards for shear testing of CMC fibers.

#### 9.4.2.3.4 *Interphase*

There are no national or international standards for shear testing of CMC interphases.

#### 9.4.2.3.5 *Overcoat*

There are no national or international standards for shear testing of CMC overcoats.

#### 9.4.2.4 *Flexure*

Several flexural test methods exist for CMCs and their constituents. These include monotonic flexural and shear strength (ambient and elevated temperatures). Eventually they will include cyclic fatigue both at ambient and elevated temperatures as well as creep. The following sections contain specifics about the test methods required to qualify materials for inclusion in MIL-HDBK-17.

##### 9.4.2.4.1 *Bulk CMC*

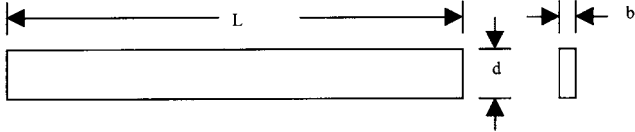
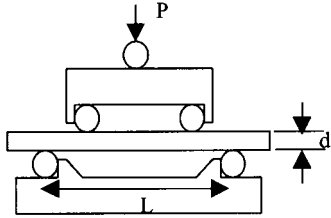
The following list of test methods and corresponding tables contain specific test parameters to be used for flexural testing bulk CMCs.

##### 9.4.2.4.1.1 *Monotonic flexural strength (ambient temperature)*

Monotonic flexural strength (ambient temperature): ASTM C 1341-95 "Standard Test Method for Flexural Properties of Continuous Fiber-Reinforced Advanced Ceramics" (see Table 9.4.2.4.1.1).



**TABLE 9.4.2.4.1.1** ASTM C1341-95 "Standard Test Method for Flexural Properties of Continuous Fiber-Reinforced Advanced Ceramics" (Ambient Temperatures).

<p><b>1. Outline:</b>                  Object Material: continuous fibre reinforced ceramic composite                  Test Method: in-plane monotonic flexural strength                  Test Environment: ambient temperature, ambient air                  Number of Test Specimens: minimum of 10 valid tests</p>	 <p>Fig. 2 Example of test specimen</p>
<p><b>2. Apparatus:</b>                  Testing Machine shall conform to the requirements of ASTM Practice E 4</p> 	<p><b>4. Test Parameters for Mil-Hdbk-17:</b>                  None specified at this time</p>
<p>Fig. 1 Schematic illustration of a flexure fixture                  Testing fixtures: i) semi-articulating for test specimens with parallelism tolerance of 0.02 mm or 0.5 % whichever is greater OR ii) fully articulating for test specimens that do not meet this requirement</p>	<p><b>5. Calculations:</b>                  3-point geometry                  Flexural stress, <math>\sigma</math> (MPa): <math>\sigma = \frac{3PL}{2bd^2}</math> where <math>\sigma</math> = maximum tensile stress in the outer fibers at a given load, <math>P</math> = load at a given point in the test (N), <math>L</math> = outer support span (mm), <math>d</math> = test specimen thickness (average or at point of break) (mm), <math>b</math> = test specimen width (average or at center) (mm)                  Flexural strain, <math>\epsilon</math> (m/m): <math>\epsilon = \frac{6Dd}{L^2}</math> where <math>\epsilon</math> = maximum strain in the outer fibers at a given load (mm/mm), <math>D</math> = deflection at beam center at a given point in the test (mm), <math>L</math> = outer support span (mm), <math>d</math> = test specimen thickness (average or at point of break) (mm)                  Flexural strength, <math>S_u</math> (MPa): <math>S_u = \frac{3LP_u}{2bd^2}</math> where <math>P_u</math> = maximum flexure load (N)                  Strain at flexural strength, <math>\epsilon_u</math> (m/m): <math>\epsilon_u = \frac{6dD_u}{L^2}</math> where <math>D_u</math> = deflection at beam center at maximum load (mm)                  Fracture strength, <math>S_F</math> (MPa): <math>S_F = \frac{3LP_F}{2bd^2}</math> where <math>P_F</math> = flexure breaking load (N)                  Strain at fracture strength, <math>\epsilon_F</math> (mm/mm): <math>\epsilon_F = \frac{6dD_F}{L^2}</math> where <math>D_F</math> = deflection at beam center at fracture load (mm),                  Tangent modulus of elasticity, <math>E</math> (MPa): <math>E = \frac{mL^3}{4bd^3}</math> where <math>m</math> = slope of tangent to the initial straight-line portion of the load-deflection</p>
<p><b>3. Procedure:</b>                  Test specimen dimensions (see Fig. 2): measure thickness &amp; width on at least three different cross sectional planes in the gage section                  Crosshead speed: 3-point and 4-point-1/4 point: <math>D = 0.167 \epsilon \frac{L^2}{d}</math>                  4-point-1/3: <math>D = 0.185 \epsilon \frac{L^2}{d}</math>                  where <math>D</math> = rate of crosshead motion (mm/s), <math>\epsilon</math> = desired strain rate (mm/mm s) (<math>1000 \times 10^{-6}</math> is recommended), <math>L</math> = outer support span (mm), <math>d</math> = test specimen thickness (mm)                  Mark test specimen for top and front surfaces                  Preload test specimen to no greater than 5 % of breaking strength                  Record load vs. displacement or stress vs. strain                  Measure and report fracture location relative to midpoint                  Record mode of fracture                  Valid test? crushing or shear failure at loading points? failure within inner span on 4-point bend test or within 2 mm of 3-point load application point?</p>	

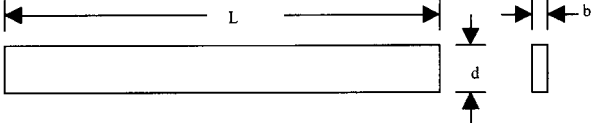
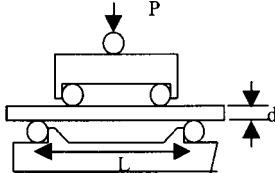
**TABLE 9.4.2.4.1.1** ASTM C1341-95 "Standard Test Method for Flexural Properties of Continuous Fiber-Reinforced Advanced Ceramics" (Ambient Temperatures) (contd.).

<p>4-point geometry, II-A</p> <p>Flexural stress, <math>\sigma</math> (MPa): <math>\sigma = \frac{3PL}{4bd^2}</math> where <math>\sigma</math> = maximum tensile stress in the outer fibers at a given load (MPa), P = load at a given point in the test (N), L = outer support span (mm), d = test specimen thickness (average or at point of break) (mm), b = test specimen width (average or at center) (mm)</p> <p>Flexural strain, <math>\epsilon</math> (m/m): <math>\epsilon = \frac{4.36Dd}{L^2}</math> where <math>\epsilon</math> = maximum strain in the outer fibers at a given load (m/m), D = deflection at beam center at a given point in the test (mm)</p> <p>Flexural strength, <math>S_u</math> (MPa): <math>S_u = \frac{3LP_u}{4bd^2}</math> where <math>P_u</math> = maximum flexure load (N)</p> <p>Strain at flexural strength, <math>\epsilon_u</math> (mm/mm): <math>\epsilon_u = \frac{4.36dD_u}{L^2}</math> where <math>D_u</math> = deflection at beam center at maximum load (mm)</p> <p>Fracture strength, <math>S_F</math> (MPa): <math>S_F = \frac{3LP_F}{4bd^2}</math> where <math>P_F</math> = breaking flexure load (N)</p> <p>Strain at fracture strength, <math>\epsilon_F</math> (mm/mm): <math>\epsilon_F = \frac{4.36dD_F}{L^2}</math> where <math>D_F</math> = deflection at beam center at fracture load (mm)</p> <p>Tangent modulus of elasticity, E (MPa): <math>E = \frac{0.17mL^3}{4bd^3}</math> where m = slope of tangent to the initial straight-line portion of the load-deflection curve (N/mm)</p> <p>4-point geometry, II-B</p> <p>Flexural stress, <math>\sigma</math> (MPa): <math>\sigma = \frac{PL}{bd^2}</math> where <math>\sigma</math> = maximum stress in the outer fibers at a given load (MPa), P = load at a given point in the test (N), L = outer support span (mm), d = test specimen thickness (average or at point of break) (mm), b = test specimen width (average or at center) (mm)</p> <p>Flexural strain, <math>\epsilon</math> (m/m): <math>\epsilon = \frac{4.70Dd}{L^2}</math> where <math>\epsilon</math> = maximum strain in the outer fibers at a given load (mm/mm), D = deflection at beam center at a given point in the test (mm)</p>	<p>Flexural strength, <math>S_u</math> (MPa): <math>S_u = \frac{LP_u}{bd^2}</math> where <math>P_u</math> = maximum flexure load (N)</p> <p>Strain at flexural strength, <math>\epsilon_u</math> (mm/mm): <math>\epsilon_u = \frac{4.70dD_u}{L^2}</math> where <math>D_u</math> = deflection at beam center at maximum load (mm)</p> <p>Fracture strength, <math>S_F</math> (MPa): <math>S_F = \frac{LP_F}{bd^2}</math> where <math>P_F</math> = breaking flexure load (N)</p> <p>Strain at fracture strength, <math>\epsilon_F</math> (mm/mm): <math>\epsilon_F = \frac{4.70dD_F}{L^2}</math> where <math>D_F</math> = deflection at beam center at fracture load (mm)</p> <p>Tangent modulus of elasticity, E (MPa): <math>E = \frac{0.21mL^3}{4bd^3}</math> where m = slope of tangent to the initial straight-line portion of the load-deflection curve (N/mm)</p> <p>Mean: <math>\bar{X} = \frac{\sum_{i=1}^n x_i}{n}</math>, deviation: s.d. = <math>\sqrt{\frac{\sum_{i=1}^n (x_i - \bar{X})^2}{n-1}}</math></p> <p><b>6. Report:</b></p> <p>Test Set:</p> <ul style="list-style-type: none"> <li>• date and location of testing</li> <li>• number of test specimens and geometry</li> <li>• type, configuration: test machine, strain measurement equipment, grip interface, load train couplers</li> <li>• conditions: temperature, relative humidity, atmosphere, test mode, load geometry (3-pt, 4 pt-1/4, 4 pt-1/3)</li> <li>• mean, standard deviation, coefficient of variation for following properties: flexural stress, strain at flexural strength, flexural strength, fracture strength, strain at fracture strength, elastic modulus, proportional limit stress, strain at proportional limit stress</li> </ul> <p>Individual Test Specimens:</p> <ul style="list-style-type: none"> <li>• overall test specimen dimensions, average cross-sectional dimensions, average</li> <li>• surface roughness</li> <li>• failure mode/location</li> <li>• flexural stress, strain at flexural strength, flexural strength, fracture strength, strain at fracture strength, elastic modulus, proportional limit stress, strain at proportional limit stress, plot of stress-strain curve</li> </ul>
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*9.4.2.4.1.2 Monotonic flexural strength (elevated temperature)*

Monotonic flexural strength (elevated temperature): ASTM C 1341-95 "Standard Test Method for Flexural Properties of Continuous Fiber-Reinforced Advanced Ceramics" (see Table 9.4.2.4.1.2).

**TABLE 9.4.2.4.1.2** ASTM C1341-95 "Standard Test Method for Flexural Properties of Continuous Fiber-Reinforced Advanced Ceramics" (Elevated Temperature).

<p><b>1. Outline:</b>                  Object Material: continuous fibre reinforced ceramic composite                  Test Method: in-plane monotonic flexural strength                  Test Environment: elevated temperatures, ambient air                  Number of Test Specimens: minimum of 10 valid tests</p>	 <p style="text-align: center;">Fig. 2 Example of flexure specimen</p>
<p><b>2. Apparatus:</b>                  Testing machine shall conform to the requirements of ASTM Practice E 4</p>  <p style="text-align: center;">Fig. 1 Schematic illustration of a flexure fixture</p> <p>Testing fixtures: i) semi-articulating for test specimens with parallelism tolerance of 0.02 mm or 0.5 % whichever is greater OR ii) fully articulating for test specimens that do not meet this requirement</p>	
<p><b>3. Procedure:</b>                  Test specimen dimensions (see Fig. 2): measure thickness &amp; width on at least three different cross sectional planes in the gage section                  Temperature rate: Linearly raise temperature in furnace within a period of 20+/-5 min. Overshoot shall not exceed 5 K for &lt; 773 °C and 1% for &gt; 773 °C for 15 s stabilize test temperature for 20+/-5 min.</p> <p>Crosshead speed: 3-point and 4-point-1/4 point: <math>\dot{D}=0.167 \dot{\epsilon} \frac{L^2}{d}</math></p> <p style="padding-left: 40px;">4-point-1/3: <math>\dot{D}=0.185 \dot{\epsilon} \frac{L^2}{d}</math></p> <p>where <math>\dot{D}</math> = rate of crosshead motion (mm/s), <math>\dot{\epsilon}</math> = desired strain rate (mm/mm s) (<math>1000 \times 10^{-6}</math> is recommended), <math>L</math> = outer support span (mm), <math>d</math> = test specimen thickness (mm)</p> <p>Mark test specimen for top and front surfaces                  Preload test specimen to no greater than 5 % of breaking strength                  Record load vs. displacement or stress vs. strain                  Measure and report fracture location relative to midpoint                  Record mode of fracture                  Valid test? crushing or shear failure at loading points? failure within inner span on 4-point bend test or within 2 mm of 3-point load application point?</p>	<p><b>4. Test Parameters for Mil-Hdbk-17:</b>                  None specified at this time</p> <p><b>5. Calculations:</b>                  3-point geometry</p> <p>Flexural stress, <math>\sigma</math> (MPa): <math>\sigma = \frac{3PL}{2bd^2}</math> where <math>\sigma</math> = maximum tensile stress in the outer fibers at a given load, <math>P</math> = load at a given point in the test (N), <math>L</math> = outer support span (mm), <math>d</math> = test specimen thickness (average or at point of break) (mm), <math>b</math> = test specimen width (average or at center) (mm)</p> <p>Flexural strain, <math>\epsilon</math> (m/m): <math>\epsilon = \frac{6Dd}{L^2}</math> where <math>\epsilon</math> = maximum strain in the outer fibers at a given load (mm/mm), <math>D</math> = deflection at beam center at a given point in the test (mm), <math>L</math> = outer support span (mm), <math>d</math> = test specimen thickness (average or at point of break) (mm)</p> <p>Flexural strength, <math>S_u</math> (MPa): <math>S_u = \frac{3LP_u}{2bd^2}</math> where <math>P_u</math> = maximum flexure load (N)</p> <p>Strain at flexural strength, <math>\epsilon_u</math> (m/m): <math>\epsilon_u = \frac{6dD_u}{L^2}</math> where <math>D_u</math> = deflection at beam center at maximum load (mm)</p> <p>Fracture strength, <math>S_F</math> (MPa): <math>S_F = \frac{3LP_F}{2bd^2}</math> where <math>P_F</math> = flexure breaking load (N)</p> <p>Strain at fracture strength, <math>\epsilon_F</math> (mm/mm): <math>\epsilon_F = \frac{6dD_F}{L^2}</math> where <math>D_F</math> = deflection at beam center at fracture load (mm),</p> <p>Tangent modulus of elasticity, <math>E</math> (MPa): <math>E = \frac{mL^3}{4bd^3}</math> where <math>m</math> = slope of tangent to the initial straight-line portion of the load-deflection</p>

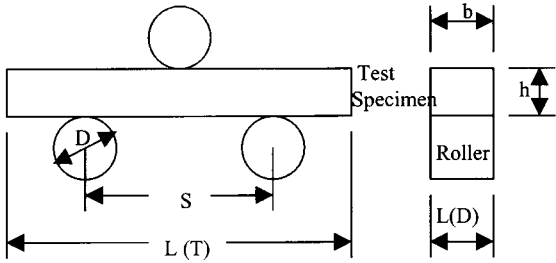
**TABLE 9.4.2.4.1.2** ASTM C1341-95 "Standard Test Method for Flexural Properties of Continuous Fiber-Reinforced Advanced Ceramics" (Elevated Temperature (contd.)).

<p>4-point geometry, II-A</p> <p>Flexural stress, <math>\sigma</math> (MPa): <math>\sigma = \frac{3PL}{4bd^2}</math> where <math>\sigma</math> = maximum tensile stress in the outer fibers at a given load (MPa), P = load at a given point in the test (N), L = outer support span (mm), d = test specimen thickness (average or at point of break) (mm), b = test specimen width (average or at center) (mm)</p> <p>Flexural strain, <math>\epsilon</math> (m/m): <math>\epsilon = \frac{4.36Dd}{L^2}</math> where <math>\epsilon</math> = maximum strain in the outer fibers at a given load (m/m), D = deflection at beam center at a given point in the test (mm)</p> <p>Flexural strength, <math>S_u</math> (MPa): <math>S_u = \frac{3LP_u}{4bd^2}</math> where <math>P_u</math> = maximum flexure load (N)</p> <p>Strain at flexural strength, <math>\epsilon_u</math> (mm/mm): <math>\epsilon_u = \frac{4.36dD_u}{L^2}</math> where <math>D_u</math> = deflection at beam center at maximum load (mm)</p> <p>Fracture strength, <math>S_F</math> (MPa): <math>S_F = \frac{3LP_F}{4bd^2}</math> where <math>P_F</math> = breaking flexure load (N)</p> <p>Strain at fracture strength, <math>\epsilon_F</math> (mm/mm): <math>\epsilon_F = \frac{4.36dD_F}{L^2}</math> where <math>D_F</math> = deflection at beam center at fracture load (mm)</p> <p>Tangent modulus of elasticity, E (MPa): <math>E = \frac{0.17mL^3}{4bd^3}</math> where m = slope of tangent to the initial straight-line portion of the load-deflection curve (N/mm)</p>	<p>Fracture strength, <math>S_F</math> (MPa): <math>S_F = \frac{LP_F}{bd^2}</math> where <math>P_F</math> = breaking flexure load (N)</p> <p>Strain at fracture strength, <math>\epsilon_F</math> (mm/mm): <math>\epsilon_F = \frac{4.70dD_F}{L^2}</math> where <math>D_F</math> = deflection at beam center at fracture load (mm)</p> <p>Tangent modulus of elasticity, E (MPa): <math>E = \frac{0.21mL^3}{4bd^3}</math> where m = slope of tangent to the initial straight-line portion of the load-deflection curve (N/mm)</p> <p>Mean: <math>\bar{X} = \frac{\sum_{i=1}^n x_i}{n}</math>, deviation: s.d. = <math>\sqrt{\frac{\sum_{i=1}^n (x_i - \bar{X})^2}{n-1}}</math></p>
<p>4-point geometry, II-B</p> <p>Flexural stress, <math>\sigma</math> (MPa): <math>\sigma = \frac{PL}{bd^2}</math> where <math>\sigma</math> = maximum stress in the outer fibers at a given load (MPa), P = load at a given point in the test (N), L = outer support span (mm), d = test specimen thickness (average or at point of break) (mm), b = test specimen width (average or at center) (mm)</p> <p>Flexural strain, <math>\epsilon</math> (m/m): <math>\epsilon = \frac{4.70Dd}{L^2}</math> where <math>\epsilon</math> = maximum strain in the outer fibers at a given load (mm/mm), D = deflection at beam center at a given point in the test (mm)</p> <p>Flexural strength, <math>S_u</math> (MPa): <math>S_u = \frac{LP_u}{bd^2}</math> where <math>P_u</math> = maximum flexure load (N)</p> <p>Strain at flexural strength, <math>\epsilon_u</math> (mm/mm): <math>\epsilon_u = \frac{4.70dD_u}{L^2}</math> where <math>D_u</math> = deflection at beam center at maximum load (mm)</p>	<p><b>6. Report:</b></p> <p>Test Set:</p> <ul style="list-style-type: none"> <li>• date and location of testing</li> <li>• number of test specimens and geometry</li> <li>• type, configuration: test machine, strain measurement equipment, grip interface, load train couplers</li> <li>• conditions: temperature, relative humidity, atmosphere, test mode, load geometry (3-pt, 4 pt-1/4, 4 pt-1/3)</li> <li>• mean, standard deviation, coefficient of variation for following properties: flexural stress, strain at flexural strength, flexural strength, fracture strength, strain at fracture strength, elastic modulus, proportional limit stress, strain at proportional limit stress</li> </ul> <p>Individual Test Specimens:</p> <ul style="list-style-type: none"> <li>• overall test specimen dimensions, average cross-sectional dimensions, average</li> <li>• surface roughness</li> <li>• failure mode/location</li> <li>• flexural stress, strain at flexural strength, flexural strength, fracture strength, strain at fracture strength, elastic modulus, proportional limit stress, strain at proportional limit stress, plot of stress-strain curve</li> </ul>

*9.4.2.4.1.3 Monotonic shear strength (ambient temperature)*

Monotonic shear strength (ambient temperature): CEN ENV 658-5 "Shear Strength (3-point) of Continuous Fiber Reinforced Ceramic Composites" (see Table 9.4.2.4.1.3).

**TABLE 9.4.2.4.1.3 CEN ENV 658-5 “Shear Strength (3-point) of Continuous Fiber-Reinforced Ceramic Composites”.**

<p><b>1. Outline:</b>                  Object Material: continuous fibre reinforced ceramic composite                  Test Method: short span bend test                  Test Environment: ambient air at room temperature                  Number of Test Specimens: minimum of 5 valid test results</p>	 <p style="text-align: center;">Figure 3 Schematic illustration of Test Fixture</p>														
<p><b>2. Apparatus:</b>                  The test machine shall conform to grade 1 in EN 10002-2 and be able to record the force                  Micrometers with accuracy of 0.1 mm and in accordance with ISO 9611                  Test fixture shall have central mandrel and outer support rollers capable of rolling outwards, mandrel and support shall have a diameter, D, of 3 mm and a length, L(D) no less than the width, b, of the test specimens (see Fig. 1)                  Test specimen shall conform to the dimensions in Table 1</p> <p style="text-align: center;">Table 1 Test Specimen Dimensions</p> <table border="1" data-bbox="449 769 1041 927"> <thead> <tr> <th>Measurement</th> <th>Dimensions (mm)</th> <th>Tolerance (mm)</th> </tr> </thead> <tbody> <tr> <td>Total Length, L(T)</td> <td>20 to 25</td> <td>+/- 1</td> </tr> <tr> <td>Thickness, h</td> <td>3</td> <td>+/- 0.1</td> </tr> <tr> <td>Width, b</td> <td>10</td> <td>+/- 0.1</td> </tr> <tr> <td>Parallelism of machined parts</td> <td></td> <td>0.05 to 0.1</td> </tr> </tbody> </table>		Measurement	Dimensions (mm)	Tolerance (mm)	Total Length, L(T)	20 to 25	+/- 1	Thickness, h	3	+/- 0.1	Width, b	10	+/- 0.1	Parallelism of machined parts	
Measurement	Dimensions (mm)	Tolerance (mm)													
Total Length, L(T)	20 to 25	+/- 1													
Thickness, h	3	+/- 0.1													
Width, b	10	+/- 0.1													
Parallelism of machined parts		0.05 to 0.1													
<p><b>4. Test Parameters for Mil-Hdbk-17:</b>                  None specified at this time</p>															
<p><b>3. Procedure:</b></p> <ul style="list-style-type: none"> <li>- Test specimen dimensions: Measure the width and thickness to the nearest 0.1 mm in the centre and each end of the test specimen</li> <li>- Measure the support span to the nearest 0.1 mm</li> </ul> <p>Crosshead speed: 0.1 mm/min or sufficient to complete test within 15 s</p> <ul style="list-style-type: none"> <li>- Install test specimen in the test machine and set crosshead speed, record force vs. time</li> <li>- Verify failure location and failure mode</li> </ul> <p>- Test validity: following circumstances will invalidate a test</p> <ul style="list-style-type: none"> <li>failure to specify and record test conditions</li> <li>failure to note mode and location of fracture</li> <li>test specimen failure not occurring within a distance from the mid-point of +/- h/4</li> </ul>	<p><b>5. Calculations:</b></p> <p>Interlaminar shear strength in plane 12, ILLS (MPa): <math>ILSS(b) = \frac{3F}{4bh}</math>                  where F = shear failure force (N), b = mean test specimen width (mm), h mean test specimen thickness (mm)</p>														
	<p><b>6. Report:</b></p> <ul style="list-style-type: none"> <li>Name and address of testing establishment</li> <li>Date of test, unique identification of report</li> <li>Customer name, address and signatory</li> <li>Reference to this standard</li> <li>Description of test material, type of fibre, batch number, date of receipt</li> <li>Area and orientation of test specimens</li> <li>Number of tests performed and number of valid tests</li> <li>Force and time record</li> <li>Interlaminar shear strength; individual results and mean</li> <li>Mode of failure and failure location</li> <li>Comments about test and/or test results</li> </ul>														

#### 9.4.2.4.2 *Matrix*

There are no national or international standards for flexural testing of CMC matrices.

#### 9.4.2.4.3 *Fiber*

There are no national or international standards for flexural testing of CMC fibers.

#### 9.4.2.4.4 *Interphase*

There are no national or international standards for flexural testing of CMC interphases.

#### 9.4.2.4.5 *Overcoat*

There are no national or international standards for flexural testing of CMC overcoats.

#### 9.4.2.5 *Fracture*

There are no national or international standards for fracture of bulk CMCs or their constituents. Eventually test methods will include those for in-plane, interlaminar, time-dependent and crack growth fracture at both ambient and elevated temperatures.

##### 9.4.2.5.1 *Bulk CMC*

There are no national or international standards for fracture testing of bulk CMCs.

##### 9.4.2.5.2 *Matrix*

There are no national or international standards for fracture testing of CMC matrices.

##### 9.4.2.5.3 *CMC fiber*

There are no national or international standards for fracture testing of CMC fibers.

##### 9.4.2.5.4 *CMC interphase*

There are no national or international standards for fracture testing of CMC interphases.

##### 9.4.2.5.5 *CMC overcoats*

There are no national or international standards for fracture testing of CMC overcoats.

### 9.4.3 **Physical**

Physical test methods include those which relate directly to those attributes which directly influence the non-thermal or mechanical aspects of a material and are inherent to the material. In particular, density, electrical, elastic constants, volume fraction, and dimensional aspects are highlighted.

#### 9.4.3.1 *Density*

The only national or international standards for density of CMCs has been developed for bulk CMCs and fibers.



9.4.3.1.1 *Bulk CMC*

The only national or international standard for density of bulk CMCs is CEN ENV 1389 "Density of Continuous Fiber Reinforced Ceramic Composites" (see Table 9.4.3.1.1).

TABLE 9.4.3.1.1 CEN ENV 1389 "Density of Continuous Fiber Reinforced Ceramic Composites".

<p><b>1. Outline:</b>  Object Material: continuous fibre reinforced ceramic matrix composite  Test Method: geometric, liquid displacement  Test Environment: ambient air, distilled water  Number of Test Specimens: individual test specimens</p>	<p>-  Remove test specimen from wire and weigh wire in liquid  - Measure temperature of liquid  - Remove test specimen from liquid, sponge with absorbent cloth  - Weigh test specimen in air</p>
<p><b>2. Apparatus:</b>  The following apparatus are needed to employ the two methods of density measurement  Method A) Drying oven, balance, calibrated measuring device (caliper with accuracy of 0.01 mm or micrometer), desiccator  Method B) Drying oven, balance, degreased metal wire dia &lt; 0.15 mm, evacuating equipment, thermometer, glass beaker, desiccator, manometer, absorbent cloth, distilled water</p>	<p><b>4. Test Parameters for Mil-Hdbk-17:</b>  None specified at this time</p>
<p><b>3. Procedure:</b>  Method A) [Geometric]  - Test specimens shall be greater than 2 g and each dimension shall be greater than 3 mm  - Dry test specimens in the oven at 283 K +/- 5 K until two successive weighings after 2 h differ by no more than 0.03 %  - Place in desiccator and allow to cool to room temperature  - Measure mass in ambient air  - Measure dimensions in at least three positions for each direction to an accuracy of 0.001 mm  - Reject any test specimen with a dimension that differs from the mean by more than 1%  Method B) [Liquid Displacement a.k.a. Archimedes]  - Test specimens shall be greater than 2 g and each dimension shall be greater than 3 mm  - Dry test specimens in the oven at 283 K +/- 5 K until two successive weighings after 2 hr differ by no more than 0.03 %  - Place in desiccator and allow to cool to room temperature  - Measure mass in ambient air  - Place cool and dry test piece within airtight vessel, evacuate to pressure less than 2500 Pa, maintain for at least 15 min  - Introduce test specimen to immersion liquid until covered by about 20 mm of liquid  - Suspend test specimen by thin wire from load pan and weigh while completely submerged</p>	<p><b>5. Calculations:</b>  Bulk density, <math>\rho</math> (g/cm<sup>3</sup>): <math>\rho = \frac{m}{v}</math> where m = mass (g), v = volume (cm<sup>3</sup>)  Dry test piece mass, <math>m_1</math> (g)  Apparent mass, <math>m_2</math> (g): <math>m_2 = m_s - m_w</math> where <math>m_s</math> = submerged mass (g), <math>m_w</math> = mass wire (g)  Soaked test piece mass, <math>m_3</math> (g)  Bulk density, <math>\rho_b</math> (g/cm<sup>3</sup>): <math>\rho_b = \frac{m_1}{m_3 - m_2} \rho_1</math> where <math>\rho_1</math> = density of liquid (g/cm<sup>3</sup>)  Apparent porosity, <math>\pi_a</math> (%) = <math>\pi_a = \frac{m_3 - m_1}{m_3 - m_2} \times 100</math></p>
	<p><b>6. Report:</b>  Name of testing establishment  Date of test/report, identification and number, operator, signatory  Reference to this standard and method used  Description of test material, manufacturer, type, batch number  Number of test pieces  Shape and surface condition of test pieces, as-fired or machined  For Method B; pressure, immersion liquid and temperature, soaking time  Individual values and mean value of bulk density and apparent porosity  Comments about test and/or test results</p>

#### 9.4.3.1.2 *Matrix*

There are no national or international standards for density of CMC matrices.

#### 9.4.3.1.3 *Fiber*

The only national or international standard for density of CMC fibers has been developed by CEN. An overview of ENV 1007-2 "Linear Mass of Fibers for Continuous Fiber Reinforced Ceramic Composites" along with specific test parameters for qualifying materials is contained in Table 9.4.3.1.3.

**TABLE 9.4.3.1.3** CEN ENV 1007-2 "Linear Mass of Fibers for Continuous Fiber-Reinforced Ceramic Composites".

<p><b>1. Outline:</b>                  Object Material: ceramic fibres; silicon carbide, silicon nitride, silicon carbon-nitride, alumino-silicate, alumina or silicon oxides                  Test Method: linear mass measurement                  Test Environment: ambient air at room temperature                  Number of Test Specimens: at least 3 test specimens</p>	<p><b>6. Report:</b>                  Name and address of testing establishment                  Date of test, unique identification of report                  Customer name, address and signatory                  Reference to this standard                  Description of test material, type of fibre, batch number, date of receipt                  Linear density of each test piece                  Comments about test and/or test results</p>
<p><b>2. Apparatus:</b>                  Cutting device                  Balance                  Plastic gloves</p>	
<p><b>3. Procedure:</b>                  Place ceramic yarn in the cutting device and cut to defined measured length                  Apply pre-tension (about 5 +/- 2.5 mN/tex) and cut to defined measured length                  Cut as many pieces per individual measurement until a minimum weighed mass of 0.1 g is achieved</p>	
<p><b>4. Test Parameters for Mil-Hdbk-17:</b>                  None specified at this time</p>	
<p><b>5. Calculations:</b>                  Linear density: <math>T_t = \frac{m}{L} \times 10^3</math>   <math>T_t</math> = linear density (g/m), m = mass of test specimen (g), L = length of test specimen (m)</p>	

#### 9.4.3.1.4 *Interphase*

There are no national or international standards for density of CMC interphases.

#### 9.4.3.1.5 *Overcoat*

There are no national or international standards for density of CMC overcoats.

#### 9.4.3.2 *Electrical*

There are no national or international standards for electrical properties of bulk CMCs or their constituents.

##### 9.4.3.2.1 *Bulk CMC*

There are no national or international standards for electrical properties of bulk CMCs.

##### 9.4.3.2.2 *Matrix*

There are no national or international standards for electrical properties of CMC matrices.

##### 9.4.3.2.3 *Fiber*

There are no national or international standards for electrical properties of CMC fibers.

##### 9.4.3.2.4 *Interphase*

There are no national or international standards for electrical properties of CMC interphases.

##### 9.4.3.2.5 *Overcoat*

There are no national or international standards for electrical properties of CMC overcoats.

#### 9.4.3.3 *Elastic constants*

There are no national or international standards for elastic constants of bulk CMCs or their constituents.

##### 9.4.3.3.1 *Bulk CMC*

There are no national or international standards for elastic constants of bulk CMCs.

##### 9.4.3.3.2 *Matrix*

There are no national or international standards for elastic constants of CMC matrices.

##### 9.4.3.3.3 *Fiber*

There are no national or international standards for elastic constants of CMC fibers.

##### 9.4.3.3.4 *Interphase*

There are no national or international standards for elastic constants of CMC interphases.

Volume 5, Part C Testing

9.4.3.3.5 *Overcoat*

There are no national or international standards for elastic constants of CMC overcoats.

9.4.3.4 *Volume fraction*

The only national or international standard for volume fraction of fibers in CMCs has been developed for bulk CMCs.

9.4.3.4.1 *Bulk CMC*

The only national or international standard for volume fraction of fibers is CMCs CEN ENV 1007-1 "Size Level of Fibers for Continuous Fiber Reinforced Ceramic Composites" (see Table 9.4.3.4.1).

**TABLE 9.4.3.4.1** CEN ENV 1007-1 "Size Level of Fibers for Continuous Fiber Reinforced Ceramic Composites".

<p><b>1. Outline:</b>  Object Material: ceramic fibres; silicon carbide, silicon nitride, silicon carbon-nitride, alumino-silicate, alumina or silicon oxides  Test Method: size content by mass  Test Environment: liquid solvent  Number of Test Specimens: at least 3 test specimens</p>	<p><b>6. Report:</b>  Name and address of testing establishment  Date of test, unique identification of report  Customer name, address and signatory  Reference to this standard  Description of test material, type of fibre, batch number, date of receipt  Size content of each test piece  Comments about test and/or test results</p>
<p><b>2. Apparatus:</b>  Balance, hot air oven, desiccator, thimble, sintered glass filter, tweezers, cutting blade, reflux extractor with condenser, heating device, boiling flask, organic solvent</p>	
<p><b>3. Procedure:</b>  Dry filter paper and thimble for 1 h at 378 K +/- 5 K  Weigh dried filter paper and test specimen  Place test specimen and filter paper into thimble  Fill boiling flask with a suitable solvent  Adjust volume of solvent to ensure there is a sufficient amount to fill reflux system  Put thimble, filter paper and test specimen into extractor and fit to boiling flask  Adjust rate of reflux to 5 cycles/h and the number of hours to ensure that complete extraction of the size is achieved  Remove thimble with filter paper after last refluxing  Dry filter paper with test specimen in hot air oven at a minimum temperature of 5 K over the boiling point of the solvent  Allow the filter paper to cool in a dessicator and weigh to nearest 0.1 mg</p>	
<p><b>4. Test Parameters for Mil-Hdbk-17:</b>  None specified at this time</p>	
<p><b>5. Calculations:</b>  Size content: <math>\tau = \frac{M_2 - (M_3 - M_2)}{M_1} \times 100</math>  <math>\tau</math> = size content, <math>M_1</math> = weight of test specimen (g), <math>M_2</math> = weight of dried filter paper (g), <math>M_3</math> = weight of fluxed filter paper (g)</p>	

#### 9.4.3.5 *Dimensions*

The only national or international standards for dimensions of CMCs has been developed for fibers.

##### 9.4.3.5.1 *Matrix (grain size)*

There are no national or international standards for grain size of CMC matrices.

##### 9.4.3.5.2 *Fiber (diameter)*

The only national or international standard for diameter of CMC fibers is CEN ENV 1007-3 "Filament Diameter of Fibers for Continuous Fiber Reinforced Ceramic Composites" (see Table 9.4.3.5.2).



**TABLE 9.4.3.5.2** CEN ENV 1007-3 "Filament Diameter of Fibers for Continuous Fiber Reinforced Ceramic Composites".

<p><b>1. Outline:</b>  Object Material: ceramic single filament  Test Method: Method A longitudinal profile, Method B transverse section, Method C laser interferometry  Test Environment: ambient air  Number of Test Specimens: per filament or fibre</p>	<p><b>4. Test Parameters for Mil-Hdbk-17:</b>  None specified at this time</p>
<p><b>2. Apparatus:</b>  Microscope with light source, eyepiece with two orthogonal wires and a double wire parallel to one of the directions, 1000x magnification  Optical microscope with photographic equipment, planimeter or image analyzer, resin  Low powered laser, vertical support, projection screen, graduated rule</p>	<p><b>5. Calculations:</b>  Filament diameter, <math>d</math> (<math>\mu\text{m}</math>):  Method A: <math>d = \frac{N_R}{2n}</math> where <math>N_R</math> = number of graduations. <math>n</math> = calibration constant  Method B: <math>d = 2\sqrt{\frac{S}{\pi}}</math> where <math>S</math> = cross-sectional area (<math>\mu\text{m}^2</math>)  Method C: <math>d = \frac{\lambda D}{i}</math> where <math>\lambda</math> = wave length (<math>\mu\text{m}</math>), <math>D</math> = distance between test specimen and screen (mm), <math>i</math> = interfringe distance (mm)</p>
<p><b>3. Procedure:</b>  <u>Method A</u>  - Mount several small lengths of ceramic fibre between microscope slide and cover slip  - Scan selected fibre with light beam  - Focus graticule and rotate eyepiece to observe the double wire parallel to ceramic fibre  - Focus on filament and bring double wire to coincide successively with the two sides of the image  - Read number or graduations  <u>Method B</u>  - Choose a resin which permit s good adhesion between the ceramic fibre and matrix  - Position the test specimen so that the fibre is perpendicular to the face of the resin block  - Pour fibre sample and polymerize at a known temperature-time cycle  - Select magnification between 1000 X and 1500 X  - Measure cross-sectional area of each filament by planimetry using either photograph of image analyzer  - Measure surface area of each filament image  <u>Method C</u>  - Use mounting tab for preparation of the filament  - Place small amount of adhesive on end of filament to bond to mounting tab slots  - Place mounting tab between laser and screen  - Measure distance from specimen to screen (<math>\geq 500</math> mm)  - Measure the interfringe distance to 0.5 mm using graduated rule</p>	<p><b>6. Report:</b>  Name and address of testing establishment  Date of test, unique identification of report  Customer name, address and signatory  Reference to this standard  Description of test material, type of fibre, batch number, date of receipt  Method of measurement selected  Type of diameter measured  Rules of sampling or sampling planes used if any  Number of filaments/fibres measured  Mean filament diameter and range  Where required, mean cross-sectional area and range  Comments about test and/or test results</p>

**9.4.4 Chemical Properties**

Reserved for future use

**9.4.5 Electrical Properties**

Reserved for future use

**9.4.6 Environmental Testing**

Reserved for future use

## **10 EVALUATION OF REINFORCEMENTS**

Reserved for future use.

### **10.1 INTRODUCTION**

Reserved for future use.

### **10.2 MECHANICAL PROPERTIES**

Reserved for future use.

#### **10.2.1 Elastic (Poisson's Ratio, modulus)**

Reserved for future use.

#### **10.2.2 Strength (FT, RT)**

Reserved for future use.

#### **10.2.3 Creep/creep rupture**

Reserved for future use.

#### **10.2.4 Fatigue**

Reserved for future use.

### **10.3 THERMAL PROPERTIES**

Reserved for future use.

#### **10.3.1 Expansion**

Reserved for future use.

#### **10.3.2 Conductivity**

Reserved for future use.

#### **10.3.3 Environmental (corrosion, erosion, wear, etc.)**

Reserved for future use.

#### **10.3.4 Oxidation**

Reserved for future use.

## **11 EVALUATION OF MATRIX MATERIALS**

Reserved for future use.

## **11.1 INTRODUCTION**

Reserved for future use.

## **11.2 MECHANICAL PROPERTIES**

Reserved for future use.

### **11.2.1 Elastic (Poisson's Ratio, modulus)**

Reserved for future use.

### **11.2.2 Strength (HT, RT)**

Reserved for future use.

### **11.2.3 Creep/creep rupture**

Reserved for future use.

### **11.2.4 Fatigue**

Reserved for future use.

## **11.3 THERMAL PROPERTIES**

Reserved for future use.

### **11.3.1 Expansion**

Reserved for future use.

### **11.3.2 Conductivity**

Reserved for future use.

### **11.3.3 Environmental (corrosion, erosion, wear, etc.)**

Reserved for future use.

### **11.3.4 Oxidation**

Reserved for future use.

### **11.3.5 Other physical (powder or preform char.)**

Reserved for future use.

## **12 EVALUATION OF INTERFACE MATERIAL**

Reserved for future use.

## **13 EVALUATION OF COMPOSITES**

Reserved for future use.

### **13.1 INTRODUCTION**

Reserved for future use.

### **13.2 MECHANICAL PROPERTIES**

#### **13.2.1 Elastic (Poisson's Ratio, modulus)**

Reserved for future use.

#### **13.2.2 Strength (HT, RT) ILT/ILS**

Reserved for future use.

#### **13.2.3 Creep/creep rupture**

Reserved for future use.

#### **13.2.4 Fatigue**

Reserved for future use.

#### **13.2.5 Open-hole tension/compression strength (notch sensitivity)**

Reserved for future use.

#### **13.2.6 Interfacial shear properties**

Reserved for future use.

### **13.3 ENVIRONMENTAL PROPERTIES**

Reserved for future use.

#### **13.3.1 Thermal expansion**

Reserved for future use.

#### **13.3.2 Conductivity**

Reserved for future use.

#### **13.3.3 Environmental (corrosion, erosion, wear, salt fog, etc.)**

Reserved for future use.

**13.3.4 Environmental effects (oxidation, corrosion, etc. )**

Reserved for future use.

**13.3.5 Oxidation**

Reserved for future use.

**13.4 REACTIONS AT THE INTERFACE (DEBONDING, DIFFUSION, ETC.) (7.9)**

Reserved for future use.

**13.5 THERMAL SHOCK RESISTANCE**

Reserved for future use.

**13.6 ELECTRICAL PROPERTIES**

Reserved for future use.

**13.7 DIELECTRIC PROPERTIES**

Reserved for future use.

**13.8 IMPACT RESISTANCE**

Reserved for future use.

**13.9 STATIC AND DYNAMIC FATIGUE**

Reserved for future use.

**13.10 PROPORTIONAL LIMIT**

Reserved for future use.

**13.11 INTERLAMINAR SHEAR PROPERTIES**

Reserved for future use.

**13.12 STRAIN AT FRACTURE**

Reserved for future use.

### **13.13 STRESS-STRAIN CURVES**

Reserved for future use.

## **14 SUBCOMPONENT TESTING – OVERVIEW OF PROBLEM**

Reserved for future use.

### **14.1 INTRODUCTION**

Reserved for future use.

### **14.2 JOINT TESTING**

Reserved for future use.

#### **14.2.1 Definitions**

Reserved for future use.

#### **14.2.2 Failure modes**

Reserved for future use.

#### **14.2.3 Thermal effects**

Reserved for future use.

#### **14.2.4 Joint configurations**

Reserved for future use.

#### **14.2.5 Design requirements**

Reserved for future use.

#### **14.2.6 Material bearing strength**

Reserved for future use.

#### **14.2.7 Open-hole tension/compression strength**

Reserved for future use.

#### **14.2.8 Thermal-mechanical fatigue strength**

Reserved for future use.

#### **14.2.9 Creep and stress rupture**

Reserved for future use.

**14.2.10 Fastener qualification tests**

Reserved for future use.

**14.3 TUBES**

Reserved for future use.

**15 MACHINING & GRINDING**

Reserved for future use.

**15.1 INTRODUCTION**

Reserved for future use.

**15.2 MACHINING CONSIDERATIONS**

Reserved for future use.

**15.3 TOOLING REQUIREMENTS**

Reserved for future use.

**15.4 SPECIMEN PREPARATION**

Reserved for future use.



**DEPARTMENT OF DEFENSE  
HANDBOOK**

**CERAMIC MATRIX COMPOSITES**

**VOLUME 5**

**PART D. DATA REQUIREMENTS AND DATA SETS**

## **PART D. DATA REQUIREMENTS AND DATA SETS**

### **16 DATA SUBMISSION, FORMAT AND REQUIREMENTS**

#### **16.1 INTRODUCTION**

Reserved for future use.

#### **16.2 PURPOSE**

The purpose of this section is to call for the minimum essential elements of information on the pedigree, composition, structure, and processing of a ceramic matrix composite to support the initial development of a CMC data base. It is meant to support the performance property data base elements.

#### **16.3 FORMAT AND UNITS**

This form is developmental and is not meant to be a fully comprehensive description of the ceramic composite. Rather, it is meant as a working tool for the first development efforts in building a CMC data base schema. The specific items in this list were developed by screening, prioritizing, and reducing the 500 data elements in the original descriptor list.

The data base elements are organized into six sets:

- A. Composite Descriptors
- B. Matrix Descriptors
- C. Reinforcement Descriptors
- D. Interphase Descriptor
- E. Architecture Descriptor
- F. Processing Descriptor
- G. External Coating Descriptor

The information items needed for each set fall into 3 groups – specimen identification (name, source, data, etc.), key technical data (compositions, structure, density, architecture, etc.), and classification data. The classification data are key elements in building a format descriptor.

**DATA SUBMISSION FORMS  
FOR CONTINUOUS FIBER CERAMIC COMPOSITES**

**In Support of Mil Handbook- 17 Ceramic Matrix Composite Data submission  
March 01 Version (STG-Gateway)**

This EXCEL spread sheet is a form for submitting mechanical property data for continuous fiber ceramic composites for the Mil Handbook 17 data collection

**Individuals and organizations submitting CMC mechanical property data to Mil Handbook 17 should complete the forms to the greatest degree possible. However, proprietary data is not necessary. And in some cases, data requests are not applicable, because of material or test limitations.**

**For data submission, refer to the handbook data submission webpage,  
<http://www.mil17.org/submit.htm>**

The spread sheet is divided into eight pages [Hot Links Provided](#)

- [Introduction Page](#)
- [Submission Information Page -- Mandatory Data](#)
- [Composite Material Description Page -- Mandatory Data](#)
- [Tensile Data Page](#)
- [Flexure Data Page](#)
- [Compression Data Page](#)
- [Inter-Laminar Shear Data Page](#)
- [In-Plane Shear Data Page](#)

If you have technical questions, points of contacts may be found at <http://www.mil17.org/coordination.htm>

The composite material description page is divided into seven description sections

- A. Composite Descriptors
- B. Matrix Descriptors
- C. Reinforcement Descriptors
- D. Interphase Descriptor
- E. Architecture Descriptor
- F. Processing Descriptor
- G. External Coating Descriptor

The test data pages are divided into six data sections

- A. Source Description
- B. Test Equipment and Procedures
- C. Specimen Description
- D. Summary Test Results
- E. Individual Test Results
- F. Stress Strain Data Curves

end

**MIL-HANDBOOK DATA SUBMISSION FORM**

**Submission Data**

[Go To Intro Page](#)

This section provides for submission data in three groups --source information, material data, and test data type/quality.

- [A. Source Information](#) *Hot Links Provided*
- [B. Material Data](#)
- [C. Test Data Type and Quality](#)

**Complete the data fields in the three groups, using all available, non-proprietary data.**

**Group A -- Source Information**

<u>ID #1-</u>	<u>ID Name</u>	<u>Format</u>	<u>Data Fields</u>
A - 1	Submission Date/s	Date	
A - 2	Source Organization	Alphanumeric	
A - 3	Source Location (City/State)	Alphanumeric	
A - 4	Contact Name	Alphanumeric	
A - 5	Contact Phone Number	Alphanumeric	
A - 6	Contact E-Mail	Alphanumeric	

**Key Question --**

**Is the test/material data in this submission export control restricted ?**

**Yes, No, Don't Know-->**

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**Group B -- Material Data**

<u>ID #1-</u>	<u>ID Name</u>	<u>Format</u>	<u>Data Fields</u>
B - 1	Material Class	Alphanumeric	Ceramic Matrix Composite
B - 2	Commercial Name/ Designation	Alphanumeric	
B - 3	Composite Description by Manufacturer	Alphanumeric	
B - 4	Manufacturer & Location	Alphanumeric	
B - 5	Manufacturer Lot/ID Number	Alphanumeric	
B - 6	Year/Date of Manufacture	Alphanumeric	
B - 7	Reinforcement Composition Class (Choose One) --	Alumina, Boron, Carbon/Graphite, Metal Carbides, Metal Silicides, Mullite, Oxide Ceramics, Quartz, S-Glass, Silicon Carbide, Silicon Nitride, Silicon-Oxy-Carbonitride, Other-Specify	
B - 8	Reinforcement Structure Class -- (Choose One)	Continuous Fiber; Discontinuous, song Fiber; Discontinuous, short fiber; Milled Fiber; Particulate, Platelet, Pulp Fiber, Sphere, Staple Fiber, Whisker, Other-Specify	
B - 9	Matrix Composition Class -- (Choose One)	Alumina, Alumino-silicate, Carbon/Graphite, Mullite, Silicon Carbide, Silicon Nitride, Silicon Carbonitride, Silicon Carboxide, Zirconia, Silica, Other-Specify	
B - 10	Fiber Interphase/Coating Composition Class (Choose One) --	None, Boron Nitride, Carbon-Graphite, Silicon Carbide, Metal Carbides, Metal Nitride, Metal Silicides, Oxide Ceramics, Multilayer, Refractory Metals, Other-Specify	
B - 11	Architecture Class -- (Choose One)	Particulate, Whisker, Fiber 1-D, Fiber 2-D, Fiber 3-D, Other-Specify	
B - 12	Symmetry Description -- (Choose One)	Quasi-Isotropic In-Plane, Isotropic, Transversely Isotropic, Orthotropic, Triaxial-Biaxial, Not Applicable, Other-Specify	
B - 13	Composite/Matrix Fabrication Class-- (Choose One)	Chemical Vapor Infiltration, Directed Metal Oxidation, Hot-Press, Preceramic Polymer, Sinter, Sol-Gel, Melt-Infiltration, Other-Specify	
B - 14	External Coating Composition Class -- (Choose One)	None, Silicon Carbide, Glass, Oxide, Carbide, Nitride, Silicide, Layered Compositions, Porcelain/ Enamel, Refractory Metals, Other-Specify	
B - 15	Developmental/Production Status-- (Choose One)	Commercially Available(CA), Limited Availability(LA), Prototype(P), Experimental development.(XD)	
B - 16	Density (g/cc)	Floating Point ( 3 Sig. Fig)	
B - 17	Porosity/Void Fraction (%)	Floating Point (3 Sig. Fig)	
B - 18	Reinf. Volume Fraction (%)	Floating Point (3 Sig. Fig.)	
B - 19	Other Comments	Alphanumeric	

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continued on next page

**Group C -- Test Data Type and Quality**

<u>ID #1-</u>	<u>Submitted Data</u>	<u>Format</u>	<u>Data Fields</u>
<b>Tensile Test</b>			
C- 1	Data Submitted	Yes - No	
C- 2	Test Temperature	Numeric - degrees C	
C- 3	Data Quality	Class A (>20 data points), Class B (6-20 data points), Class C(<6 data points) per sample group.	
<b>Flexure Test</b>			
C- 4	Data Submitted	Yes - No	
C- 5	Test Temperature	Numeric - degrees C	
C- 6	Data Quality	Class A (>20 data points), Class B (6-20 data points), Class C(<6 data points) per sample group.	
<b>Compression Test</b>			
C- 7	Data Submitted	Yes - No	
C- 8	Test Temperature	Numeric - degrees C	
C- 9	Data Quality	Class A (>20 data points), Class B (6-20 data points), Class C(<6 data points) per sample group.	
<b>Interlaminar Shear Test</b>			
C- 10	Data Submitted	Yes - No	
C- 11	Test Temperature	Numeric - degrees C	
C- 12	Data Quality	Class A (>20 data points), Class B (6-20 data points), Class C(<6 data points) per sample group.	
<b>In-Plane Shear Test</b>			
C- 13	Data Submitted	Yes - No	
C- 14	Test Temperature	Numeric - degrees C	
C- 15	Data Quality	Class A (>20 data points), Class B (6-20 data points), Class C(<6 data points) per sample group.	

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end

**MIL-HANDBOOK DATA SUBMISSION FORM**

**Composite Material Description**

[Go To Intro Page](#)

This section provides for basic descriptions of the composite material divided into seven data groups.

- [A. Composite Descriptors](#)
- [B. Matrix Descriptors](#)
- [C. Reinforcement Descriptors](#)
- [D. Interphase Descriptor](#)

- [E. Architecture Descriptor](#)
- [F. Processing Descriptor](#)
- [G. External Coating Descriptor](#)

*Hot Links Provided*

**Complete the data fields in the seven groups, using all available, non-proprietary data.**

**Group A -- COMPOSITE DESCRIPTORS**

<u>ID#2-</u>	<u>ID Name</u>	<u>Format</u>	<u>Data Fields</u>
A - 1	Material Class	Alphanumeric	Ceramic Matrix Composite
A - 2	Commercial Name/ Designation	Alphanumeric	
A - 3	Composite Description by Manufacturer	Alphanumeric	
A - 4	Manufacturer & Location	Alphanumeric	
A - 5	Manufacturer Lot/ID Number	Alphanumeric	
A - 6	Year/Date of Manufacture	Alphanumeric	
A - 7	Reinforcement Composition Class (Choose One) --	Alumina, Boron, Carbon/Graphite, Metal Carbides, Metal Silicides, Mullite, Oxide Ceramics, Quartz, S-Glass, Silicon Carbide, Silicon Nitride, Silicon-Oxy-Carbonitride, Other-Specify	
A - 8	Reinforcement Structure Class -- (Choose One)	Continuous Fiber; Discontinuous, long Fiber; Discontinuous, short fiber; Milled Fiber; Particulate, Platelet, Pulp Fiber, Sphere, Staple Fiber, Whisker, Other-Specify	
A - 9	Matrix Composition Class (Choose One) --	Alumina, Alumino-silicate, Carbon/Graphite, Mullite, Silicon Carbide, Silicon Nitride, Silicon Carbonitride, Silicon Carboxide, Zirconia, Silica, Other-Specify	
A - 10	Fiber Interphase/Coating Composition Class -- (Choose One)	None, Boron Nitride, Carbon-Graphite, Silicon Carbide, Metal Carbides, Metal Nitride, Metal Silicides, Oxide Ceramics, Multilayer, Refractory Metals, Other-Specify	
A - 11	Architecture Class -- (Choose One)	Particulate, Whisker, Fiber 1-D, Fiber 2-D, Fiber 3-D, Other-Specify	
A - 12	Symmetry Description -- (Choose One)	Quasi-Isotropic In-Plane, Isotropic, Transversely Isotropic, Orthotropic, Triaxial-Biaxial, Not Applicable, Other-Specify	
A - 13	Composite/Matrix Fabrication Class-- (Choose One)	Chemical Vapor Infiltration, Directed Metal Oxidation, Hot-Press, Preceramic Polymer, Sinter, Sol-Gel, Melt-Infiltration, Other-Specify	
A - 14	External Coating Composition Class -- (Choose One)	None, Silicon Carbide, Glass, Oxide, Carbide, Nitride, Silicide, Layered Compositions, Porcelain/ Enamel, Refractory Metals, Other-Specify	
A - 15	Developmental/Production Status-- (Choose One)	Commercially Available(CA), Limited Availability(LA), Prototype(P), Experimental development.(XD)	
A - 16	Density (g/cc)	Floating Point ( 3 Sig. Fig)	g/cc
A - 17	Porosity/Void Fraction (%)	Floating Point ( 3 Sig. Fig)	%
A - 18	Reinf. Volume Fraction (%)	Floating Point ( 3 Sig. Fig)	%
A - 19	Service/Exposure History	Alphanumeric	
A - 20	Report/Publication Source	Alphanumeric	
A - 21	Report/Publication Reference	Alphanumeric	
A - 22	Report/Publication Date	Alphanumeric	

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**Group B -- MATRIX DESCRIPTORS**

<u>ID#2-</u>	<u>ID Name</u>	<u>Format</u>	<u>Data Fields</u>
B- 1	Common/Commercial Name	Alphanumeric	
B- 2	Additional Name Information	Alphanumeric	
B- 3	Manufacturer & Location	Alphanumeric	
B- 4	Manufacturer Lot/ID Number	Alphanumeric	
B- 5	Year/Date of Manufacture	Alphanumeric	
B- 6	Major Constituents & Fractions	Alphanumeric	
B- 7	Major Phases & Fractions	Alphanumeric	
B- 8	Developmental/Production Status -- (Choose One)	Commercially Available(CA), Limited Availability(LA), Prototype(P), Experimental Development.(XD)	
B- 9	Other Comments	Alphanumeric	

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**Group C -- REINFORCEMENT DESCRIPTORS**

<u>ID#2-</u>	<u>ID Name</u>	<u>ID# &amp; Format</u>	<u>Data Fields</u>
C-1	Common/Commercial Name	Alphanumeric	
C-2	Additional Name Information	Alphanumeric	
C-3	Manufacturer & Location	Alphanumeric	
C-4	Manufacturer Lot/ID Number	Alphanumeric	
C-5	Year/Date of Manufacture	Alphanumeric	
C-6	Fiber Diameter / Particulate Size (micron)	Floating Point (2 Sig. Fig)	micron
C-7	Major Chemical Constituents & Fractions	Alphanumeric	
C-8	Major Phases & Fractions	Alphanumeric	
C-9	<b>Filler/Particulate</b> - Method Of Manufacture -- (Choose One)	Filler/Particulate -- Mine, Mill, & Size; Solid-State Reaction; Liquid Phase Reaction; Gas-Phase Reaction; Other-Specify)	
C-10	<b>Fiber</b> - Method Of Manufacture - (Choose One)	Chemical Vapor Deposition (CVD); Melt Spin; Sol-Gel Spin & Heat Treat; Inorganic Polymer Spin & Heat Treat; Particulate Consolidation & Sinter; Other-Specify)	
C-11	Nominal Fiber Tensile Strength		MPa
C-12	Developmental/Production Status (Choose One)	Commercially Available(CA), Limited Availability(LA), Prototype(P), Experimental Development.(XD)	

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**Group D -- INTERPHASE/COATING DESCRIPTORS**

<u>ID#2-</u>	<u>ID Name</u>	<u>ID# &amp; Format</u>	<u>Data Fields</u>
D-1	Common/Commercial Name	Alphanumeric	
D-2	Additional Name Information	Alphanumeric	
D-3	Manufacturer & Location	Alphanumeric	
D-4	Manufacturer Lot/ID Number	Alphanumeric	
D-5	Year/Date of Manufacture	Alphanumeric	
D-6	Total Coating Thickness (microns)	Floating Point (3 Sig. Fig)	micron
D-7	Major Constituents & Fractions	Alphanumeric	
D-8	Major Phases & Fractions	Alphanumeric	
D-9	Interphase/Coating Structure - (Choose One)	Single Layer, Multilayer, Porous, Microcracked, Multiphase, Fugitive, Other-Specify	
D-10	Interphase/Coating Method Of Manufacture -- (Choose One)	Chemical Vapor Deposition, In-Situ Reaction, Gas Phase Reaction, Liquid Coating, Particulate Coating, Other-Specify	
D-11	Developmental/Production Status(Choose One)	Commercially Available(CA), Limited Availability(LA), Prototype(P), Experimental Development.(XD)	
D-12			

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Volume 5, Part D Data Requirements and Data Sets

**Group E -- ARCHITECTURE DESCRIPTORS**

<b>ID#2-</b>	<b>ID Name</b>	<b>Format</b>	<b>Data Fields</b>
E-1	Common/Commercial Name	Alphanumeric	
E-2	Additional Name Information	Alphanumeric	
E-3	Manufacturer & Location	Alphanumeric	
E-4	Manufacturer Lot/ID Number	Alphanumeric	
E-5	Year/Date of Manufacture	Alphanumeric	
E-6	Architecture Class -- (Choose One)	Particulate, Whisker, 1-D Tape, 1-D Winding, 2-D Weaving, 3-D Weaving, 2-D Braiding, 3-D Braiding, Other-Specify	
E-7	Symmetry Description (Choose One)	Quasi-Isotropic In-Plane, Isotropic, Transversely Isotropic, Orthotropic, Triaxial-Biaxial, Not Applicable, Other-Specify	
E-8	Ply Count (if appl)	Integer	
E-9	Per-Ply Thickness (mm)	Floating Point (3 Sig. Fig)	mm
E-10	Lay-Up Sequence/Code (if appl)	Alphanumeric	
E-11	End/Tow/Roving/Yarn Description	Alphanumeric	
E-12	Thread Count in the Major Directions (1, 2, 3) (x/inch)	Integer	#/inch
E-13	Weave Description (Choose One)	Plain, Crowsfoot, 5-harness satin, 8-harness atin, 12-harness satin, Other-Specify	
E-14	Working Form For Composite Fabrication -- (Choose One)	Dry Tow, Impregnated Tow, Non-Woven Mat, Dry Fabric, Prepreg - Tape Or Fabric, Dry Preform, Other-Specify	
E-15	Other Comments	Alphanumeric	
E-16	Developmental/Production Status(Choose One)	Commercially Available(CA), Limited Availability(LA), Prototype(P), Experimental Development.(XD)	

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**Group F -- PROCESSING/FABRICATION DESCRIPTORS**

<b>ID#2-</b>	<b>ID Name</b>	<b>Format</b>	<b>Data Fields</b>
F-1	Common/Commercial Name	Alphanumeric	
F-2	Additional Name Information	Alphanumeric	
F-3	Manufacturer & Location	Alphanumeric	
F-4	Manufacturer Lot/ID Number	Alphanumeric	
F-5	Composite/Matrix Fabrication Class-- (Choose One)	Chemical Vapor Infiltration, Directed Metal Oxidation, Hot-Press, Preceramic Polymer, Sinter, Sol-Gel, Melt-Infiltration, Other-Specify	
F-6	Forming/Shaping Method (Choose One) -	Preform Shaping, Cold/Warm Die Pressing, Lamination, Resin Transfer Molding, Injection Molding, Vacuum Bagging, Slip Casting, Other-Specify	
F-7	Developmental/Production Status(Choose One)	Commercially Available(CA), Limited Availability(LA), Prototype(P), Experimental Development.(XD)	
F-8	Prepreg Name (if appl.)	Alphanumeric	
F-9	Prepreg Source & Location (if appl.)	Alphanumeric	
F-10	Prepreg. Lot/ID Number (if appl.)	Alphanumeric	
F-11	Prepreg. Dev./Product. Status (Choose One) -	Commercially Available(CA), Limited Availability(LA), Prototype(P), Experimental Development.(XD)	
F-12	Final Machining Condition	Alphanumeric	
F-13	Maximum Fabrication Temperature	Integer	C
F-14	Other Comments	Alphanumeric	

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**Group G -- EXTERNAL COATING DESCRIPTORS (if applicable)**

<b>ID#2-</b>	<b>ID Name</b>	<b>Format</b>	<b>Data Fields</b>
G-1	Common/Commercial Name	Alphanumeric	
G-2	Additional Name Information	Alphanumeric	
G-3	Manufacturer Name & Location	Alphanumeric	
G-4	Manufacturer Lot/ID Number	Alphanumeric	
G-5	External Coating Composition Class -- (Choose One)	None, Silicon Carbide, Glass, Oxide, Carbide, Nitride, Silicide, Layered Compositions, Porcelain/ Enamel, Refractory Metals, Other-Specify	
G-6	Year/Date of Manufacture	Alphanumeric	
G-7	Total Coating Thickness (mm)	Floating Point (3 Sig. Fig)	mm
G-8	Major Constituents & Fractions	Alphanumeric	
G-9	Major Phases & Fractions	Alphanumeric	
G-10	# of Layers	Integer	
G-11	External Coating Method Of Manufacture -- (Choose One)	Chemical Vapor Deposition, In-Situ Reaction, Liquid Coating, Particulate Coating, Pack Cementation, Other-Specify	
G-12	External Coating Structure (Choose One) --	Single Layer, Multilayer, Porous, Multiphase, Other-Specify,	
G-13	Other Comments	Alphanumeric	
G-14	Developmental/Production Status(Choose One)	Commercially Available(CA), Limited Availability(LA), Prototype(P), Experimental Development.(XD)	

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**MIL-HANDBOOK DATA SUBMISSION FORM**

**Tensile Data**

[Go To Intro Page](#)

This section provides the format for submission of tensile data on test source, test method, test specimen, and test results. This data format follows ASTM C1278.

The test data is divided into six groups --

[A. Source Description](#)

*Hot Links Provided*

[B. Test Equipment and Procedures](#)

[C. Specimen Description](#)

[D. Summary Test Results](#)

[E. Individual Test Results](#)

[F. Stress Strain Data Curves](#)

**Complete the data fields in the six groups using all available, non-proprietary data**

**Group A -- SOURCE DESCRIPTION**

<u>ID# 3-</u>	<u>ID Name</u>	<u>Format</u>	<u>Data Fields</u>
A - 1	Test Date/s	Date	
A - 2	Test Organization	Alphanumeric	
A - 3	Test Location (City/State)	Alphanumeric	
A - 4	Contact Name	Alphanumeric	
A - 5	Contact Phone Number	Alphanumeric	
A - 6	Contact E-Mail	Alphanumeric	
A - 7	Report/Publication Reference	Alphanumeric	
A - 8	Report/Publication Date	Alphanumeric	
A - 9	Organization Run ID	Alphanumeric	

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**Group B -- TENSILE TEST EQUIPMENT AND PROCEDURES DESCRIPTORS**

<u>ID# 3-</u>	<u>ID Name</u>	<u>Format</u>	<u>Data Fields</u>
B- 1	Tensile Test Standard Specification	Alphanumeric	
B- 2	Test machine description/ identification	Alphanumeric	
B- 3	Test Control Method— (Choose one)	Load, displacement, strain	
B- 4	Testing Rate [mm/min., N/min., or %/min.]	Alphanumeric	mm/min
B- 5	Grip method— (Choose one)	face loaded, edge loaded, pin loaded	
B- 6	Grip Manufacturer/Model Number	Alphanumeric	
B- 7	Grip Pressure [ Pa, psi]	Flt.Pt.(3 sig. Fig)	Pa
B- 8	Load Train Coupler Manufacturer/Model	Alphanumeric	
B- 9	Coupler Type - (Choose One)	Rigid, self aligning	
B- 10	Strain Measurement System- Manufacturer/Model Number	Alphanumeric	
B- 11	Strain measurement method—(Choose one)	Clip-on, strain gage, capacitive, laser flags, laser interferometry	
B- 12	Extensometer gage length [mm] (if applicable)	Flt.Pt.(3 sig. Fig)	mm
B- 13	Atmosphere – (Choose one)	Air, inert, combustion, Other-specif	
B- 14	Test Temperature [°C]	Flt.Pt.(3 sig. Fig)	C
B- 15	Humidity (RH%)	Flt.Pt.(2 sig. Fig)	RH%
B- 16	Time to temperature [Minutes]	Flt.Pt.(2 sig. Fig)	Minutes
B- 17	Soak time at temperature [Minutes]	Flt.Pt.(2 sig. Fig)	Minutes
B- 18	Alignment- % Bending before test	Flt.Pt.(2 sig. Fig)	
B- 19	Alignment- % Bending after test	Flt.Pt.(2 sig. Fig)	

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**Group C -- TENSILE SPECIMEN DESCRIPTORS (See Material Page for Baseline Data)**

<u>ID# 3-</u>	<u>ID Name</u>	<u>Format</u>	<u>Data Fields</u>	
C- 1	Manufacturer Serial Number	Alphanumeric		
C- 2	Sample geometry—(Choose one)	Dogbone, Straight sided		
C- 3	Gage Width [mm]	Flt.Pt.(4 sig. Fig)		mm
C- 4	Thickness [mm]	Flt.Pt.(4 sig. Fig)		mm
C- 5	Specimen Gage length [mm]	Flt.Pt.(2 sig. Fig)		mm
C- 6	Stress Orientation (Choose one)	Alphanumeric (11, 22, 33) or Angle from Major Fiber Axis		
C- 7	Specimen Preparation & Machining	Alphanumeric		
C- 8	Preconditioning/Exposure	Alphanumeric		
C- 9	Other Comments	Alphanumeric		

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**Group D -- Summary Tensile Test Data**

<u>ID# 3-</u>	<u>ID Name</u>	<u>Format</u>	Mean	Std. .Dev	Count	Max	Min
D- 1	Test Temperature [°C]	Flt. Pt. (3 Sig. Fig)					
D- 2	Tensile Strength [MPa]	Flt. Pt. (4 Sig. Fig)					
D- 3	Strain at Tensile Strength [%]	Flt. Pt. (3 Sig. Fig)					
D- 4	Fracture Strength [MPa]	Flt. Pt. (4 Sig. Fig)					
D- 5	Strain at Fracture [%]	Flt. Pt. (3 Sig. Fig)					
D- 6	Modulus of Elasticity [GPa]	Flt.Pt. (4 sig. Fig)					
D- 7	Poisson's Ratio $\nu_{12}$ (Ratio between in-plane transverse and longitudinal strain)	Flt. Pt. (3 Sig. Fig)					
D- 8	Poisson's Ratio $\nu_{13}$ (Ratio between thru-thickness transverse and longitudinal strain)	Flt. Pt. (3 Sig. Fig)					
D- 9	Proportional Limit Stress $\sigma_o$ [MPa]	Flt. Pt. (4 Sig. Fig)					
D- 10	Proportional Limit Method —(Choose one)	offset yield [OY] or extension under load [EL], Other-Specify					
D- 11	Proportional Limit variable (offset strain or strain extension value) [%]	Flt. Pt. (4 Sig. Fig)					
D- 12	Modulus of Toughness [Msi]	Flt. Pt. (4 Sig. Fig)					
D- 13	Modulus of Resilience [Msi]	Flt. Pt. (4 Sig. Fig)					

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**Group E -- Individual Specimen Tensile Test Data**

Sample #																	
Test Temperature [°C]																	
Tensile Strength [MPa]																	
Strain at Tensile Strength [%]																	
Fracture Strength [MPa]																	
Strain at Fracture [%]																	
Modulus of Elasticity [GPa]																	
Poisson's Ratio $\nu_{12}$ (Ratio between in-plane transverse and longitudinal strain)																	
Poisson's Ratio $\nu_{13}$ (Ratio between thru-thickness transverse and longitudinal strain)																	
Proportional Limit Stress $\sigma_0$ [MPa]																	
NA																	
Proportional Limit variable (offset strain or strain extension value) [%]																	
Modulus of Toughness [Msi]																	
Modulus of Resilience [Msi]																	
Fracture Type—(Choose one) <i>gage, radius, grip, thermal gradient,</i>																	
Fracture location [mm] measured from bottom of specimen gage length.																	
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Volume 5, Part D Data Requirements and Data Sets

**Group F -- Stress-Strain Data Curves**

Stress-strain data      ascii, tab delimited

Sample #	Strain(%)	Stress (Mpa)	Strain(%)	Stress (Mpa)	Strain(%)	Stress (Mpa)	Strain(%)	Stress (Mpa)	Strain(%)	Stress (Mpa)	Strain(%)	Stress (Mpa)	Strain(%)	Stress (Mpa)	Strain(%)	Stress (Mpa)
Data point 1																
Data point 2																
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**MIL-HANDBOOK DATA SUBMISSION FORM Page 4**

**Flexure Data**

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This section provides the format for submission of flexure data on test source, test method, test specimen, and test results. This data format follows ASTM C1341. The test data is divided into six groups --

- [A. Source Description](#) *Hot Links Provided*
- [B. Test Equipment and Procedures](#)
- [C. Specimen Description](#)
- [D. Summary Test Results](#)
- [E. Individual Test Results](#)
- [F. Stress Strain Data Curves](#)

**Complete the data fields in these six groups, using all available, non-proprietary data**

**Group A -- SOURCE DESCRIPTION**

<u>ID# 4-</u>	<u>ID Name</u>	<u>Format</u>	<u>Data Fields</u>
A - 1	Test Date/s	Date	
A - 2	Test Organization	Alphanumeric	
A - 3	Test Location (City/State)	Alphanumeric	
A - 4	Contact Name	Alphanumeric	
A - 5	Contact Phone Number	Alphanumeric	
A - 6	Contact E-Mail	Alphanumeric	
A - 7	Report/Publication Reference	Alphanumeric	
A - 8	Report/Publication Date	Alphanumeric	
A - 9	Organization Run ID	Alphanumeric	

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**Group B -- FLEXURE TEST EQUIPMENT AND PROCEDURES DESCRIPTORS**

<u>ID# 4-</u>	<u>ID Name</u>	<u>Format</u>	<u>Data Fields</u>
B- 1	Flexure Test Standard Specification	Alphanumeric	
B- 2	Test machine description/ identification	Alphanumeric	
B- 3	Test Control Method— (Choose one)	Load, displacement, strain	
B- 4	Testing Rate [mm/min., N/min., or %/min.]	Flt.Pt.(3 sig. Fig)	mm/min
B- 5	Load Configuration (Choose One)	3 Pt, 4 Pt-1/4 Pt, 4-Pt-1/3 Pt	
B- 6	Fixture Manufacturer/ Description	Alphanumeric	
B- 7	Fixture Type (Choose One)	Fixed, Semi-Articulated, Fully Articulated	
B- 8	Roller Bearings (Choose One)	Fixed, Rolling	
B- 9	Outer Span Length (mm)	Flt.Pt.(3 sig. Fig)	mm
B- 10	Load Span Length (mm- 4 pt only)	Flt.Pt.(3 sig. Fig)	mm
B- 11	Strain measurement method—(Choose one)	Deflectometer, strain gage	
B- 12	Strain Measurement System- Manufacturer/Model Number	Alphanumeric	
B- 13	Atmosphere – Choose one	Air, inert, combustion, Other-specify	
B- 14	Test Temperature [°C]	Flt.Pt.(3 sig. Fig)	C
B- 15	Humidity (RH%)	Flt. Point (2 Sig. Fig)	RH%
B- 16	Time to temperature [Minutes]	Flt. Point (2 sig. Fig)	minutes
B- 17	Soak time at temperature [Minutes]	Flt. Point (2 sig. Fig)	minutes

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**Group C -- FLEXURE SPECIMEN DESCRIPTORS (See Page 1 for Baseline Data)**

<u>ID# 4-</u>	<u>ID Name</u>	<u>Format</u>	<u>Data Fields</u>	
C- 1	Manufacturer Serial Number	Alphanumeric		
C- 2	Specimen Thickness [mm]	Flt. Point (4 sig. Fig)		
C- 3	Specimen Width [mm]	Flt. Point (4 sig. Fig)		mm
C- 4	Specimen Length [mm]	Flt. Point (2 sig. Fig)		mm
C- 5	Stress Orientation Plane (Choose one)	Alphanumeric (11, 22, 33) or Angle from Major Fiber Axis		
C- 6	Specimen Preparation & Machining	Alphanumeric		
C- 7	Preconditioning/Oxidation	Alphanumeric		

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**Group D -- Summary Flexure Test Data**

<u>ID# 4-</u>	<u>ID Name</u>	<u>Format</u>	Mean	Std. Dev	Count	Max	Min
D-1	Test Temperature [°C]	Flt. Pt. (3 Sig. Fig)					
D-2	Flexure Strength [MPa]	Flt.Pt. (4 sig. Fig)					
D-3	Strain at Flexure Strength [%]	Flt. Pt. (3 Sig. Fig)					
D-4	Fracture Strength [MPa]	Flt.Pt. (4 sig. Fig)					
D-5	Strain at Fracture [%]	Flt. Pt. (3 Sig. Fig)					
D-6	Modulus of Elasticity [GPa]	Flt.Pt. (4 sig. Fig)					
D-7	Proportional Limit Stress $\sigma$ [MPa]	Flt. Pt. (3 Sig. Fig)					
D-8	Proportional Limit Method —(Choose one)	offset yield [OY] or extension under load [EL]					
D-9	Proportional Limit variable (offset strain or strain extension value) [%]	Flt. Point (3 Sig. Fig)					
D-10	Modulus of Toughness [Msi]	Flt. Point (3 Sig. Fig)					

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**MIL-HANDBOOK DATA SUBMISSION FORM**

**Compression Data**

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This section provides the format for submission of compression data on test source, test method, test specimen, and test results. This data format follows ASTM C1358

The test data is divided into six groups --

- [A. Source Description](#)
- [B. Test Equip. and Procedures](#)
- [C. Specimen Description](#)
- [D. Summary Test Results](#)
- [E. Individual Test Results](#)
- [F. Stress Strain Data Curves](#)

*Hot Links Provided*

**Complete the data fields in these six groups, using all available, non-proprietary data**

**Group A -- SOURCE DESCRIPTION**

<u>ID# 5-</u>	<u>ID Name</u>	<u>ID# &amp; Format</u>	<u>Data Fields</u>
A - 1	Test Date/s	Date	
A - 2	Test Organization	Alphanumeric	
A - 3	Test Location (City/State)	Alphanumeric	
A - 4	Contact Name	Alphanumeric	
A - 5	Contact Phone Number	Alphanumeric	
A - 6	Contact E-Mail	Alphanumeric	
A - 7	Report/Publication Reference	Alphanumeric	
A - 8	Report/Publication Date	Alphanumeric	
A - 9	Organization Run ID	Alphanumeric	

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**Group B -- COMPRESSION TEST EQUIPMENT AND PROCEDURES DESCRIPTORS**

<u>ID# 5-</u>	<u>ID Name</u>	<u>Format</u>	<u>Data Fields</u>
B- 1	Compression Test Standard Specification	Alphanumeric	
B- 2	Test machine description/ identification	Alphanumeric	
B- 3	Test Control Method— (Choose one)	Load, displacement, strain	
B- 4	Testing Rate [mm/min., N/min., or %/min.]	Alphanumeric	
B- 5	Compression Fixture Description	Alphanumeric	
B- 6	Fixture Manufacturer/Model Number	Alphanumeric	
B- 7	Strain Measurement System- Manufacturer/Model Number	Alphanumeric	
B- 8	Strain measurement method—(Choose one)	Clip-on, strain gage, capacitive, laser flags, laser interferometry	
B- 9	Extensometer gage length [mm] (if applicable)	Flt. Point (3 sig. Fig)	mm
B- 10	Atmosphere – Choose one	air, inert, combustion, Other-specify	
B- 11	Test Temperature [°C]	Flt. Point (3 sig. Fig)	C
B- 12	Humidity (RH%)	Flt. Point (2 Sig. Fig)	RH%
B- 13	Time to temperature [Minutes]	Flt. Point (2 Sig. Fig)	minutes
B- 14	Soak time at temperature [Minutes]	Flt. Point (2 Sig. Fig)	minutes
B- 15	Alignment- % Bending before test	Flt. Point (2 Sig. Fig)	%
B- 16	Alignment- % Bending after test	Flt. Point (2 Sig. Fig)	%

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**Group C --COMPRESSION SPECIMEN DESCRIPTORS (See Material Page for Baseline Data)**

<u>ID# 5-</u>	<u>ID Name</u>	<u>Format</u>	<u>Data Fields</u>	
C- 1	Manufacturer Serial Number	Alphanumeric		
C- 2	Gage Width [mm]	Flt. Point (4 sig. Fig)		mm
C- 3	Gage Thickness [mm]	Flt. Point (4 sig. Fig)		mm
C- 4	Specimen Gage length [mm]	Flt. Point (4 sig. Fig)		mm
C- 5	Specimen Preparation & Machining	Alphanumeric		
C- 6	Stress Orientation (Choose one)	Alphanumeric (11, 22, 33) or Angle from Major Fiber Axis		
C- 6	Preconditioning/Exposure	Alphanumeric		
C- 7	Calculated Euler Buckling Stress (MPa)	Flt. Point (4 sig. Fig)		MPa

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**Group D -- Summary Compression Test Data**

<u>ID# 5-</u>	<u>ID Name</u>	<u>Format</u>	Mean	Std.Dev	Count	Max	Min
D-1	Test Temperature [°C]	Flt. Point (3 Sig. Fig)					
D-1	Tensile Strength [MPa]	Flt. Point (4 Sig. Fig)					
D-2	Strain at Tensile Strength [%]	Flt. Point (3 Sig. Fig)					
D-3	Fracture Strength [MPa]	Flt. Point (4 Sig. Fig)					
D-4	Strain at Fracture [%]	Flt. Point (3 Sig. Fig)					
D-5	Modulus of Elasticity [GPa]	Flt. Point (4 Sig. Fig)					
D-6	Poisson's Ratio $\nu_{12}$ (Ratio between in-plane transverse and longitudinal strain)	Flt. Point (3 Sig. Fig)					
D-7	Poisson's Ratio $\nu_{13}$ (Ratio between thru-thickness transverse and longitudinal strain)	Flt. Point (3 Sig. Fig)					
D-8	Proportional Limit Stress $\sigma$ [MPa]	Flt. Point (4 Sig. Fig)					
D-9	Proportional Limit Method —(Choose one)	offset yield [OY] or extension under load [EL]. <u>Other-Specify</u>					
D-10	Proportional Limit variable (offset strain or strain extension value) [%]	Flt. Point (4 Sig. Fig)					
D-11	Modulus of Toughness [Msi]	Flt. Point (3 Sig. Fig)					
D-12	Modulus of Resilience [Msi]	Flt. Point (3 Sig. Fig)					

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continued on next page

**SET E -- Individual Specimen Compression Test Data**

<b>Sample #</b>														
Test Temperature [°C]														
Tensile Strength [MPa]														
Strain at Tensile Strength [%]														
Fracture Strength [MPa]														
Strain at Fracture [%]														
Modulus of Elasticity [GPa]														
Poisson's Ratio v12 (Ratio between in-plane transverse and longitudinal strain)														
Poisson's Ratio v13 (Ratio between thru-thickness transverse and longitudinal strain)														
Proportional Limit Stress $\sigma$ [MPa]														
NA														
Proportional Limit variable (offset strain or strain extension value) [%]														
Modulus of Toughness [Msi]														
Modulus of Resilience [Msi]														
Fracture Type—(Choose one) <i>gage, radius, grip, thermal gradient,</i>														
Fracture location [mm] measured from bottom of specimen gage length.														

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continued on next page

**SET F -- Stress-Strain Data Curves**

Stress-strain data

ascii, tab delimited

Sample #	Strain(%)	Stress (Mpa)	Strain(%)	Stress (Mpa)	Strain(%)	Stress (Mpa)	Strain(%)	Stress (Mpa)	Strain(%)	Stress (Mpa)	Strain(%)	Stress (Mpa)
Data point 1												
Data point 2												
Data point 3												
Data point 4												
Data point 5												
Data point 6												
Data point 7												
Data point 8												
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Data point 54												
Data point 55												

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end

**MIL-HANDBOOK DATA SUBMISSION FORM**

**Interlaminar(I-L) Shear Data**

[Go To Intro Page](#)

This data format follows ASTM C1292

This section provides the format for submission of I-L shear data on test source, test method, test specimen, and test results.

The test data is divided into six groups --

- [A. Source Description](#)
- [B. Test Equip. and Procedures](#)
- [C. Specimen Description](#)
- [D. Summary Test Results](#)
- [E. Individual Test Results](#)
- [F. Force-Displacement Data Curves](#)

*Hot Links Provided*

**Complete the data fields in these six groups, using all available, non-proprietary data**

**Group A -- SOURCE DESCRIPTION**

<u>ID #6-</u>	<u>ID Name</u>	<u>ID# &amp; Format</u>	<u>Data Fields</u>
A - 1	Test Date/s	Date	
A - 2	Test Organization	Alphanumeric	
A - 3	Test Location (City/State)	Alphanumeric	
A - 4	Contact Name	Alphanumeric	
A - 5	Contact Phone Number	Alphanumeric	
A - 6	Contact E-Mail	Alphanumeric	
A - 7	Report/Publication Reference	Alphanumeric	
A - 8	Report/Publication Date	Alphanumeric	
A - 9	Organization Run ID	Alphanumeric	

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**Group B -- IL SHEAR TEST EQUIPMENT AND PROCEDURES DESCRIPTORS**

<u>ID #6-</u>	<u>ID Name</u>	<u>Format</u>	<u>Data Fields</u>
B- 1	I-L Shear Test Standard Specification	Alphanumeric	
B- 2	Test machine description/ identification	Alphanumeric	
B- 3	Test Control Method— (Choose one)	Load, displacement, strain	
B- 4	Testing Rate [mm/min., N/min., or %/min.]	Alphanumeric	
B- 5	I-L Fixture Description	Alphanumeric	
B- 6	I-L Fixture Manufacturer/ Model Number	Alphanumeric	
B- 7	Atmosphere – Choose one	air, inert, combustion, Other-specify	
B- 8	Test Temperature [°C]	Flt. Point (3 sig. Fig)	C
B- 9	Humidity (RH%)	Flt. Point (2 Sig. Fig)	RH%
B- 10	Time to temperature [Minutes]	Flt. Point (2 Sig. Fig)	min
B- 11	Soak time at temperature [Minutes]	Flt. Point (2 Sig. Fig)	min

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**Group C -- IL SHEAR SPECIMEN DESCRIPTORS (See Material Page for Baseline Data)**

<u>ID #6-</u>	<u>ID Name</u>	<u>Format</u>	<u>Data Fields</u>	
C- 1	Manufacturer Serial Number	Alphanumeric		
C- 2	Specimen Length (mm)	Flt. Point (4 sig. Fig)		mm
C- 3	Specimen Width (mm)	Flt. Point (4 sig. Fig)		mm
C- 4	Specimen Thickness (mm)	Flt. Point (4 sig. Fig)		mm
C- 5	Notch Depth (mm)	Flt. Point (2 sig. Fig)		mm
C- 6	Notch Separation (mm)	Flt. Point (2 sig. Fig)		mm
C- 7	Shear Load Orientation (Choose One)	12/21, 13/23, 13/32		
C- 8	Specimen Preparation & Machining	Alphanumeric		
C- 9	Preconditioning	Alphanumeric		

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**Group D -- Summary IL Shear Test Data**

<u>ID #6-</u>	<u>ID Name</u>	<u>Format</u>	<u>Mean</u>	<u>Std. Dev</u>	<u>Count</u>	<u>Max</u>	<u>Min</u>
D-1	Test Temperature [°C]	Flt. Point (3 Sig. Fig)					
D-2	I-L Shear Strength [MPa]	Flt. Point (4 Sig. Fig)					

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continued on next page

**SET E -- Individual Specimen IL Shear Test Data**

<b>Sample #</b>																				
Test Temperature [°C]																				
I-L Shear Strength [MPa]																				
Fracture Type Description																				

GO TO TOP of Form

**SET F --Force- Displacement Data Curves**

Stress-strain data  
ascii, tab delimited

Sample #	Displac.(mm)	Force (N)	Displac.(m)	Force (N)	Displac.(m)	Force (N)	Displac.(m)	Displac.(m)	Force (N)	Displac.(m)	Displac.(m)	Force (N)	Displac.(m)	Displac.(m)	Force (N)
Data point 1															
Data point 2															
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end

**MIL-HANDBOOK DATA SUBMISSION FORM**

**In-Plane (I-P) Shear Data**

[Go To Intro Page](#)

This section provides the format for submission of I-L shear data on test source, test method, test specimen, and test results. This data format follows ASTM C1292

The test data is divided into six groups --

- [A. Source Description](#)
- [B. Test Equip. and Procedures](#)
- [C. Specimen Description](#)
- [D. Summary Test Results](#)
- [E. Individual Test Results](#)
- [F. Force-Displacement Data Curves](#)

*Hot Links Provided*

**Complete the data fields in these six groups, using all available, non-proprietary data**

**Group A -- SOURCE DESCRIPTION**

<u>ID #7-</u>	<u>ID Name</u>	<u>Format</u>	<u>Data Fields</u>
A - 1	Test Date/s	Date	
A - 2	Test Organization	Alphanumeric	
A - 3	Test Location (City/State)	Alphanumeric	
A - 4	Contact Name	Alphanumeric	
A - 5	Contact Phone Number	Alphanumeric	
A - 6	Contact E-Mail	Alphanumeric	
A - 7	Report/Publication Reference	Alphanumeric	
A - 8	Report/Publication Date	Alphanumeric	
A - 9	Organization Run ID	Alphanumeric	

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**Group B -- IP SHEAR TEST EQUIPMENT AND PROCEDURES DESCRIPTORS**

<u>ID #7-</u>	<u>ID Name</u>	<u>Format</u>	<u>Data Fields</u>
B- 1	I-P Shear Test Standard Specification	Alphanumeric	
B- 2	Test machine description/ identification	Alphanumeric	
B- 3	Test Control Method— (Choose one)	Load, displacement, strain	
B- 4	Testing Rate [mm/min., N/min]	Alphanumeric	
B- 5	I-P Fixture Description	Alphanumeric	
B- 6	I-P Fixture Manufacturer/ Model Number	Alphanumeric	
B- 7	Atmosphere – Choose one	air, inert, combustion, Other-specify	
B- 8	Test Temperature [°C]	Flt. Point (3 sig. Fig)	C
B- 9	Humidity (RH%)	Flt. Point (2 Sig. Fig)	RH%
B- 10	Time to temperature [Minutes]	Flt. Point (2 sig. Fig)	min
B- 11	Soak time at temperature [Minutes]	Flt. Point (2 sig. Fig)	min

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**Group C -- IP SHEAR SPECIMEN DESCRIPTORS (See Material Page for Baseline Data)**

<u>ID #7-</u>	<u>ID Name</u>	<u>Format</u>	<u>Data Fields</u>	
C- 1	Manufacturer Serial Number	Alphanumeric		
C- 2	Specimen Length (mm)	Flt. Point (4 sig. Fig)		mm
C- 3	Specimen Width (mm)	Flt. Point (4 sig. Fig)		mm
C- 4	Specimen Thickness (mm)	Flt. Point (4 sig. Fig)		mm
C- 5	Notch Separation (mm)	Flt. Point (2 sig. Fig)		mm
C- 6	Notch Depth (mm)	Flt. Point (2 sig. Fig)		mm
C- 7	Specimen Preparation & Machining	Alphanumeric		
C- 8	Preconditioning	Alphanumeric		
C- 9	Shear Load Orientation (Choose One)	12/21, 13/23, 13/32		

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**Group D -- Summary IP Shear Test Data**

<u>ID #7-</u>	<u>ID Name</u>	<u>Format</u>	Mean	Std. Dev	Count	Max	Min
D-1	Test Temperature [°C]	Flt. Point (3 Sig. Fig)					
D-2	I-P Shear Strength [MPa]	Flt. Point (4 Sig. Fig)					

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continued on next page

**SET E -- Individual Specimen IP Shear Test Data**

<b>Sample #</b>																			
Test Temperature [°C]																			
I-L Shear Strength [MPa]																			
Fracture Type Description																			

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**SET F --Force- Displacement Data Curves**

Sample #	ascii, tab delimited													
	Displac.(mm)	Force (N)	Displac.(m)	Force (N)	Displac.(m)	Force (N)	Displac.(m)	Force (N)	Displac.(m)	Force (N)	Displac.(m)	Force (N)	Displac.(m)	Force (N)
Data point 1														
Data point 2														
Data point 3														
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end

## **16.4 DESIGN PROPERTIES**

Reserved for future use.

## **17 STATISTICAL METHODS**

Reserved for future use.

### **17.1 INTRODUCTION**

Reserved for future use.

#### **17.1.1 An overview of methods for calculating statistically based properties**

Reserved for future use.

#### **17.1.2 Computer software**

Reserved for future use.

#### **17.1.3 Symbols**

Reserved for future use.

#### **17.1.4 Statistical terms**

Reserved for future use.

### **17.2 BACKGROUND**

Reserved for future use.

#### **17.2.1 Statistically-based design allowables**

Reserved for future use.

#### **17.2.2 Basis values for unstructured data**

Reserved for future use.

#### **17.2.3 Basis values in the presence of batch-to-batch variability**

Reserved for future use.

#### **17.2.4 Batches, panels, and confounding**

Reserved for future use.

**17.2.5 Sample size guidelines for determining basis values**

Reserved for future use.

**17.3 CALCULATION OF STATISTICALLY BASED MATERIAL PROPERTIES**

Reserved for future use.

**17.3.1 Guide to computational procedures**

Reserved for future use.

**17.3.2 Subpopulation compatibility – structured or unstructured**

Reserved for future use.

**17.3.3 Detecting outliers**

Reserved for future use.

**17.3.4 Basis values for unstructured data**

Reserved for future use.

**17.3.5 Basis values for structured data**

Reserved for future use.

**17.3.6 Exploratory data analysis**

Reserved for future use.

**17.3.7 Examples of computational procedures**

Reserved for future use.

**17.4 MISCELLANEOUS STATISTICAL METHODS**

Reserved for future use.

**17.4.1 Confidence intervals for the coefficient of variation**

Reserved for future use.

**17.4.2 Statistical procedures for process control**

Reserved for future use.

**17.4.3 Alternate material statistical procedures**

Reserved for future use.

#### **17.4.4 Typical stress-strain curves**

Reserved for future use.

### **17.5 STATISTICAL TABLES AND APPROXIMATIONS**

Reserved for future use.

## **18 CMC PROPERTY DATA**

### **18.1 INTRODUCTION**

Reserved for future use.

#### **18.1.1 Organization of data in handbook**

Reserved for future use.

#### **18.1.2 Presentation of data**

Reserved for future use.

#### **18.1.3 Material coding and documentation**

Reserved for future use.

#### **18.1.4 Materials systems codes**

Reserved for future use.

#### **18.1.5 Material orientation codes**

Reserved for future use.

#### **18.1.6 Symbols, Abbreviations, and systems of units**

Reserved for future use.

#### **18.1.7 Definitions**

Reserved for future use.

### **18.2 CMC SYSTEMS - PROPERTY DATA**

18.2.1 CMC system #1

<b>COMPOSITE</b>			
<i>Composite Name</i>		<b>9/99 EPM SiC/SiC</b>	<i>Manufacturer</i>
<i>Composite Description</i>		<b>SiC fiber / BN-SiC / MI SiC</b>	<i>Manuf. Lot #</i>
<i>Reinforc. Vol %</i>		<b>36%</b>	<i>Date of Manufacture</i>
<i>Density</i>		<b>2.86</b>	<i>Fabrication Class</i>
<i>% Porosity</i>		<b>10%-20%</b>	<i>Dev/Prod. Status</i>
			<b>Honeywell ACI</b>
			<b>0981-01 and 0604-01</b>
			<b>Sep-99</b>
			<b>Silicon Melt Infiltration</b>
			<b>Limited availability</b>
<b>Reinforcement</b>	<i>Name</i>	<b>Sylramic</b>	<i>Manufacturer</i>
<i>Composition</i>		<b>SiC</b>	<i>Structure/Architecture</i>
<i>Ply Count</i>		<b>8</b>	<i>Ply Thickness</i>
<i>Fabric Areal Weight</i>		<b>281.58 g/ m<sup>2</sup></b>	<i>Laminate Lay-up Sequence</i>
			<b>COI</b>
			<b>5-harness satin weave fabric</b>
			<b>0.25 mm</b>
			<b>[0/90]<sub>4s</sub></b>
<b>Matrix</b>	<i>Composition</i>	SiC + Siliconized SiC	<i>Source</i>
			<b>Melt infiltration</b>
<b>Interphase /Fiber Coating</b>			<i>Composition</i>
<i>Thickness</i>		<b>0.5µm</b>	<i>Source</i>
			<b>Silicon doped boron nitride</b>
			<b>CVI</b>
<b>External Coating</b>	<i>Composition</i>	<b>na</b>	<i>Thickness</i>
<i>Fabrication Method</i>		<b>na</b>	<i>Source</i>
			<b>na</b>
<i>Date of Testing</i>		<b>11/99 to 02/00</b>	<i>Date of Data Submittal</i>
			<b>Mar-01</b>

<b>Summary Test Data</b>	Temperature	816°C	1038°C	1204°C	1315°C
		Air	Air	Air	Air
		none	none	none	none
Normalized or Measured		Measured	Measured	Measured	Measured
Elastic Modulus $E_x^t$ (GPa)	Mean	208	209	182	158
	Approval Class	S	S	S	S
Elastic Modulus $E_y^t$ (GPa)	Mean	208	209	182	158
	Approval Class	S	S	S	S
Elastic Modulus $E_z^t$ (GPa)	Mean				
	Approval Class				
Poisson's Ratio $\nu_{xy}^t$	Mean				
	Approval Class				
Poisson's Ratio $\nu_{yz}^t$	Mean				
	Approval Class				
Poisson's Ratio $\nu_{zx}^t$	Mean				
	Approval Class				
Shear Modulus $G_{xy}^s$ (GPa)	Mean				
	Approval Class				
Shear Modulus $G_{yz}^s$ (MPa)	Mean				
	Approval Class				
Shear Modulus $G_{zx}^s$ (MPa)	Mean				
	Approval Class				
Prop. Limit Stress $F^{pl}$ MPa	Mean				
	Approval Class				
Thermal Conductivity $\alpha_x$	Mean				
	Approval Class				
Thermal Conductivity $\alpha_y$	Mean				
	Approval Class				
Thermal Conductivity $\alpha_z$	Mean				
	Approval Class				
Thermal Expansion $K_x$	Mean				
	Approval Class				
Thermal Expansion $K_y$	Mean				
	Approval Class				
Thermal Expansion $K_z$	Mean				
	Approval Class				

Approval Class = F - Fully Approved, I - Interim, S - Screening

<b>COMPOSITE</b>			
<i>Composite Name</i>	<b>9/99 EPM SiC/SiC</b>	<i>Manufacturer</i>	<b>Honeywell ACI</b>
<i>Composite Description</i>	<b>SiC fiber / BN-SiC / MI SiC</b>	<i>Manuf. Lot #</i>	<b>0981-01 and 0604-01</b>
<i>Reinforc. Vol %</i>	<b>36%</b>	<i>Date of Manufacture</i>	<b>Sep-99</b>
<i>Density</i>	<b>2.86</b>	<i>Fabrication Class</i>	<b>Silicon Melt Infiltration</b>
<i>% Porosity</i>	<b>10%-20%</b>	<i>Dev/Prod. Status</i>	<b>Limited availability</b>
<b>Reinforcement</b>	<b>Sylramic</b>	<i>Manufacturer</i>	<b>COI</b>
<i>Name</i>	<b>SiC</b>	<i>Structure/Architecture</i>	<b>5-harnes satin weave fabric</b>
<i>Composition</i>	<b>8</b>	<i>Ply Thickness</i>	<b>0.25 mm</b>
<i>Ply Count</i>	<b>281.58 g/ m<sup>2</sup></b>	<i>Laminate Lay-up Sequence</i>	<b>[0<sub>i</sub>/90<sub>i</sub>]4s</b>
<i>Fabric Areal Weight</i>			
<b>Matrix</b>	<b>SiC + Siliconized SiC</b>	<i>Source</i>	<b>Melt infiltration</b>
<i>Composition</i>			
<b>Interphase /Fiber Coating</b>		<i>Composition</i>	<b>Silicon doped boron nitride</b>
<i>Thickness</i>	<b>0.5µm</b>	<i>Source</i>	<b>CVI</b>
<b>External Coating</b>	<b>na</b>	<i>Thickness</i>	<b>na</b>
<i>Composition</i>			
<i>Fabrication Method</i>	<b>na</b>	<i>Source</i>	<b>na</b>
<i>Date of Testing</i>	<b>11/99 to 02/00</b>	<i>Date of Data Submittal</i>	<b>Mar-01</b>

<b>Summary Test Data</b>	Temperature	816°C	1038°C	1204°C	1315°C
		Air	Air	Air	Air
	Atmosphere	none	none	none	none
	Precondition	Measured	Measured	Measured	Measured
	Normalized or Measured	Measured	Measured	Measured	Measured
Tensile Strength $F_x^{tu}$ (MPa)	Mean	362	325	307	295
	Approval Class	S	S	S	S
Tensile Strength $F_y^{tu}$ (MPa)	Mean	362	325	307	295
	Approval Class	S	S	S	S
Tensile Strength $F_z^{tu}$ (MPa)	Mean				
	Approval Class				
Strain $\epsilon_x^{tu}$ (µε)	Mean	4810	4350	4590	4300
	Approval Class	S	S	S	S
Strain $\epsilon_y^{tu}$ (µε)	Mean	4810	4350	4590	4300
	Approval Class	S	S	S	S
Strain $\epsilon_z^{tu}$ (µε)	Mean				
	Approval Class				
Prop. Limit Stress $F_x^{pl}$ (MPa)	Mean	177	168	166	163
	Approval Class	S	S	S	S
Prop. Limit Stress $F_y^{pl}$ (MPa)	Mean	177	168	166	163
	Approval Class	S	S	S	S
Prop. Limit Stress $F_z^{pl}$ (MPa)	Mean				
	Approval Class				
Compressive Strength $F_x^{cu}$ (MPa)	Mean				
	Approval Class				
Compressive Strength $F_y^{cu}$ (MPa)	Mean				
	Approval Class				
Compressive Strength $F_z^{cu}$ (MPa)	Mean				
	Approval Class				
In-Plane Shear Strength $F_{xy}^{su}$ (MPa)	Mean	47.2			
	Approval Class	S			
Interlaminar Shear Strength $F_{xy}^{su}$ (MPa)	Mean				
	Approval Class				
Interlaminar Shear Strength $F_{yz}^{su}$ (MPa)	Mean				
	Approval Class				
Flexure Strength $F^b$ (MPa)	Mean				
	Approval Class				

Approval Class = F - Fully Approved, I - Interim, S - Screening

<b>COMPOSITE</b>			
Composite Name	9/99 EPM SiC/SiC	Manufacturer	Honeywell ACI
Composite Description	SiC fiber / BN-SiC / MI SiC	Manuf. Lot #	0981-01 and 0604-01
Reinforc. Vol %	36%	Date of Manufacture	Sep-99
Density	2.86	Fabrication Class	Silicon Melt Infiltration
% Porosity	10%-20%	Dev/Prod. Status	Limited availability
<b>Reinforcement</b> Name	Sylramic	Manufacturer	COI
Composition	SiC	Structure/Architecture	5-harness satin weave fabric
Ply Count	8	Ply Thickness	0.25 mm
Fabric Areal Weight	281.58 g/ m <sup>2</sup>	Laminate Lay-up Sequence	[0/90 <sub>i</sub> ]4s
<b>Matrix</b> Composition	SiC + Siliconized SiC	Source	Melt infiltration
<b>Interphase /Fiber Coating</b>		Composition	Silicon doped boron nitride
Thickness	0.5µm	Source	CVI
<b>External Coating</b> Composition	na	Thickness	na
Fabrication Method	na	Source	na
<b>Tensile Test Method</b>		Test File ID#	
Test Method Spec.	ASTM C 1275-95	Date of Testing	11/99 to 02/00
Normalized by:		Load/Ext. Strain Rate	0.5% / min.
Modulus Calc Method	LS fit between 30 & 100 MPa	P.L. Calculation Method	Offset yield w/ 50µ offset

Date of Data Submittal Mar-01

Tension Test Data	Temperature	816°C		1038°C		1204°C		1315°C	
		Normalized	Measured	Normalized	Measured	Normalized	Measured	Normalized	Measured
	Atmosphere	Air		Air		Air		Air	
	Precondition	none		none		none		none	
	Gage Length	27.94 mm		27.94 mm		27.94 mm		27.94 mm	
	Stress Orientation	0°		0°		0°		0°	
Ultimate Tensile Strength F <sub>x</sub> <sup>tu</sup> (MPa)	Mean				362				325
	S.Dev.				32.2				29.2
	# of Specimens				24				6
	# of Batches				6				6
	Approval Class				S				S
Elastic Modulus E <sub>x</sub> <sup>t</sup> (GPa)	Mean				208				209
	S.Dev.				14.0				14.5
	# of Specimens				24				6
	# of Batches				6				6
	Approval Class				S				S
Prop. Limit Stress F <sub>x</sub> <sup>pl</sup> (MPa)	Mean				177				168
	S.Dev.				19.4				19.9
	# of Specimens				24				6
	# of Batches				6				6
	Approval Class				S				S
Strain at F <sup>ult</sup> ε <sub>x</sub> <sup>tu</sup> (µε)	Mean				4810				4350
	S.Dev.				626				621
	# of Specimens				24				6
	# of Batches				6				6
	Approval Class				S				S

Approval Class = F - Fully Approved, I - Interim, S - Screening



Volume 5, Part D Data Requirements and Data Sets

<b>COMPOSITE</b>			
<i>Composite Name</i>	<b>9/99 EPM SiC/SiC</b>	<i>Manufacturer</i>	<b>Honeywell ACI</b>
<i>Composite Description</i>	<b>SiC fiber / BN-SiC / MI SiC</b>	<i>Manuf. Lot #</i>	<b>0981-01 and 0604-01</b>
<i>Reinforc. Vol %</i>	<b>36%</b>	<i>Date of Manufacture</i>	<b>Sep-99</b>
<i>Density</i>	<b>2.86</b>	<i>Fabrication Class</i>	<b>Silicon Melt Infiltration</b>
<i>% Porosity</i>	<b>10%-20%</b>	<i>Dev/Prod. Status</i>	<b>Limited availability</b>
<b>Reinforcement</b> <i>Name</i>	<b>Sylramic</b>	<i>Manufacturer</i>	<b>COI</b>
<i>Composition</i>	<b>SiC</b>	<i>Structure/Architecture</i>	<b>5-harness satin weave fabric</b>
<i>Ply Count</i>	<b>8</b>	<i>Ply Thickness</i>	<b>0.25 mm</b>
<i>Fabric Areal Weight</i>	<b>281.58 g/ m<sup>2</sup></b>	<i>Laminate Lay-up Sequence</i>	<b>[0<sub>r</sub>/90<sub>r</sub>]<sub>4s</sub></b>
<b>Matrix</b> <i>Composition</i>	<b>SiC + Siliconized SiC</b>	<i>Source</i>	<b>Melt infiltration</b>
<b>Interphase /Fiber Coating</b> <i>Thickness</i>	<b>0.5µm</b>	<i>Composition</i>	<b>Silicon doped boron nitride</b>
		<i>Source</i>	<b>CVI</b>
<b>External Coating</b> <i>Composition</i>	<b>na</b>	<i>Thickness</i>	<b>na</b>
<i>Fabrication Method</i>	<b>na</b>	<i>Source</i>	<b>na</b>
<b>Interlaminar Shear Test Method</b> <i>Test Method Spec.</i>	<b>ASTM C 1292 - 95</b>	<i>Test File ID#</i>	
<i>Normalized by:</i>		<i>Notch Distance</i>	<b>6.37 mm</b>
		<i>Date of Testing</i>	<b>Feb-01</b>

<b>Shear Test Data</b>		Temperature	
		<b>816°C</b>	
		Atmosphere	
		<b>Air</b>	
		Precondition	
		<b>none</b>	
		Normalized	Measured
Ultimate Interlaminar Strength	Mean		<b>47.2</b>
	S.Dev.		<b>7.24</b>
F <sub>ZX</sub> <sup>SU</sup> (MPa)	# of Specimens		<b>24</b>
	# of Batches		<b>6</b>
		Approval Class	<b>S</b>

Approval Class = F - Fully Approved, I - Interim, S - Screening

18.2.2 CMC system #2

<b>COMPOSITE</b>			
Composite Name	Enhanced SiC/SiC	Manufacturer	Honeywell ACI
Composite Description	SiC/ Carbon / SiC	Manuf. Lot #	multiple
Reinforc. Vol %	40%	Date of Manufacture	11/94 ... 12/97
Density	2.3 g/cc	Fabrication Class	Chemical Vapor Infiltration
% Porosity	12%	Dev/Prod. Status	Commercially Available
<b>Reinforcement</b> Name	CG Nicalon™	Manufacturer	COI
Composition	SiC	Structure/Architecture	5-harness satin weave fabric
Ply Count	11	Ply Thickness	~0.3 mm
Fabric Areal Weight	~287 g/m <sup>2</sup>	Laminate Lay-up Sequence	[0/90 <sub>i</sub> ] <sub>ns</sub>
<b>Matrix</b> Composition	SiC	Source	CVI
<b>External Coating</b>		Thickness	0.1 to 0.2 mm
Composition	SiC	Source	Chemical Vapor Infiltration
Fabrication Method	Chemical Vapor Infiltration	Date of Manufacture	11/94 to 12/97

Date of Testing 11/94 to 12/97 Date of Data Submittal Mar-01

Summary Test Data	Temperature Atmosphere Precondition	23°C	850°C	1100°C	1200°C
		Air	Air	Air	Air
		none	none	none	none
	Normalized or Measured	Measured	Measured	Measured	Measured
Elastic Modulus E <sub>x</sub> <sup>t</sup> (GPa)	Mean	129	113	122	119
	Approval Class	S	S	S	S
Elastic Modulus E <sub>y</sub> <sup>t</sup> (GPa)	Mean	129	113	122	119
	Approval Class	S	S	S	S
Elastic Modulus E <sub>z</sub> <sup>t</sup> (GPa)	Mean				
	Approval Class				
Poisson's Ratio ν <sub>xy</sub> <sup>t</sup>	Mean				
	Approval Class				
Poisson's Ratio ν <sub>yz</sub> <sup>t</sup>	Mean				
	Approval Class				
Poisson's Ratio ν <sub>zx</sub> <sup>t</sup>	Mean				
	Approval Class				
Shear Modulus G <sub>xy</sub> <sup>s</sup> (GPa)	Mean				
	Approval Class				
Shear Modulus G <sub>yz</sub> <sup>s</sup> (MPa)	Mean				
	Approval Class				
Shear Modulus G <sub>zx</sub> <sup>s</sup> (MPa)	Mean				
	Approval Class				
Prop. Limit Stress F <sub>x</sub> <sup>pl</sup> MPa	Mean				
	Approval Class				
Thermal Conductivity α <sub>x</sub>	Mean				
	Approval Class				
Thermal Conductivity α <sub>y</sub>	Mean				
	Approval Class				
Thermal Conductivity α <sub>z</sub>	Mean				
	Approval Class				
Thermal Expansion K <sub>x</sub>	Mean				
	Approval Class				
Thermal Expansion K <sub>y</sub>	Mean				
	Approval Class				
Thermal Expansion K <sub>z</sub>	Mean				
	Approval Class				

Approval Class = F - Fully Approved, I - Interim, S - Screening

<b>COMPOSITE</b>			
Composite Name	Enhanced SiC/SiC	Manufacturer	Honeywell ACI
Composite Description	SiC/ Carbon / SiC	Manuf. Lot #	multiple
Reinforc. Vol %	40%	Date of Manufacture	11/94 ... 12/97
Density	2.3 g/cc	Fabrication Class	Chemical Vapor Infiltration
% Porosity	12%	Dev/Prod. Status	Commercially Available
<b>Reinforcement</b> Name	CG Nicalon™	Manufacturer	COI
Composition	SiC	Structure/Architecture	5-harness satin weave fabric
Ply Count	11	Ply Thickness	~0.3 mm
Fabric Areal Weight	~287 g/m <sup>2</sup>	Laminate Lay-up Sequence	[0/90] <sub>i</sub> ns
<b>Matrix</b> Composition	SiC	Source	CVI
<b>External Coating</b>		Thickness	0.1 to 0.2 mm
Composition	SiC	Source	Chemical Vapor Infiltration
Fabrication Method	Chemical Vapor Infiltration	Date of Manufacture	11/94 to 12/97

Date of Testing: 11/94 to 12/97      Date of Data Submittal: Mar-01

Summary Test Data	Temperature	23°C	850°C	1100°C	1200°C
		Atmosphere	Air	Air	Air
	Precondition	none	none	none	none
		Normalized or Measured	Measured	Measured	Measured
Tensile Strength $F_x^{tu}$ (MPa)	Mean	230	265	275	250
	Approval Class	S	S	S	S
Tensile Strength $F_y^{tu}$ (MPa)	Mean	230	265	275	250
	Approval Class	S	S	S	S
Tensile Strength $F_z^{tu}$ (MPa)	Mean				
	Approval Class				
Strain $\epsilon_x^{tu}$ ( $\mu\epsilon$ )	Mean	4680	5860	6090	7250
	Approval Class	S	S	S	S
Strain $\epsilon_y^{tu}$ ( $\mu\epsilon$ )	Mean	4680	5860	6090	7250
	Approval Class	S	S	S	S
Strain $\epsilon_z^{tu}$ ( $\mu\epsilon$ )	Mean				
	Approval Class				
Prop. Limit Stress $F_x^{pl}$ (MPa)	Mean	72.2	79.1	89.3	76.9
	Approval Class	S	S	S	S
Prop. Limit Stress $F_y^{pl}$ (MPa)	Mean	72.2	79.1	89.3	76.9
	Approval Class	S	S	S	S
Prop. Limit Stress $F_z^{pl}$ (MPa)	Mean				
	Approval Class				
Compressive Strength $F_x^{cu}$ (MPa)	Mean	577			
	Approval Class	S			
Compressive Strength $F_y^{cu}$ (MPa)	Mean	577			
	Approval Class	S			
Compressive Strength $F_z^{cu}$ (MPa)	Mean				
	Approval Class				
In-Plane Shear Strength $F_{xy}^{su}$ (MPa)	Mean	38.0			
	Approval Class	S			
Interlaminar Shear Strength $F_{yz}^{su}$ (MPa)	Mean				
	Approval Class				
Interlaminar Shear Strength $F_{zx}^{su}$ (MPa)	Mean				
	Approval Class				
Flexural Strength $F_x^b$ (MPa)	Mean	420			447
	Approval Class	S			S

Approval Class = F - Fully Approved, I - Interim, S - Screening

<b>COMPOSITE</b>			
Composite Name	Enhanced SiC/SiC	Manufacturer	Honeywell ACI
Composite Description	SiC/ Carbon / SiC	Manuf. Lot #	multiple
Reinforc. Vol %	40%	Date of Manufacture	11/94 ... 12/97
Density	2.3 g/cc	Fabrication Class	Chemical Vapor Infiltration
% Porosity	12%	Dev/Prod. Status	Commercially Available
<b>Reinforcement</b> Name	CG Nicalon™	Manufacturer	COI
Composition	SiC	Structure/Architecture	5-harness satin weave fabric
Ply Count	11	Ply Thickness	~0.3 mm
Fabric Areal Weight	~287 g/m <sup>2</sup>	Laminate Lay-up Sequence	[0 <sub>r</sub> /90 <sub>r</sub> ] <sub>ns</sub>
<b>Matrix</b> Composition	SiC	Source	CVI
<b>Interphase /Fiber Coating</b> Composition		Composition	Carbon
Thickness	0.5µm	Source	CVI
<b>External Coating</b> Composition	SiC	Thickness	0.1 to 0.2 mm
Fabrication Method	Chemical Vapor Infiltration	Source	Chemical Vapor Infiltration
<b>Tensile Test Method</b>	=	Test File ID#	multiple
Test Method Spec.	C1275-95 & C1359-96	Date of Testing	11/94 to 12/97
Normalized by:		Load/Ext. Strain Rate	0.5 mm / min
Modulus Calc Method	3.5 to 35.5 MPa LS fit	P.L. Calculation Method	Offset yield (0.005%)

Date of Data Submittal

Mar-01

Tension Test Data	Temperature	23°C		850°C		1100°C		1200°C	
		Normalized	Measured	Normalized	Measured	Normalized	Measured	Normalized	Measured
	Atmosphere	Air		Air		Air		Air	
	Precondition	None		None		None		None	
	Gage Length	25.4 mm		25.4 mm		25.4 mm		25.4 mm	
	Stress Orientation	0°		0°		0°		0°	
Ultimate Tensile Strength F <sub>x</sub> <sup>tu</sup> (MPa)	Mean	230		265		275		250	
	S.Dev.	20.1		10.9		10.8		9.5	
	# of Specimens	90		8		9		5	
	# of Batches	21		5		9		1	
	Approval Class								
Elastic Modulus E <sub>x</sub> <sup>t</sup> (GPa)	Mean	129		114		122		119	
	S.Dev.	13.1		3.94		12.4		4.22	
	# of Specimens	90		8		9		5	
	# of Batches	21		5		9		1	
	Approval Class								
Prop. Limit Stress F <sub>x</sub> <sup>pl</sup> (MPa)	Mean	72.2		79.1		89.3		76.9	
	S.Dev.	11.0		7.35		14.4		4.06	
	# of Specimens	90		7		9		5	
	# of Batches	21		4		9		1	
	Approval Class								
Strain at F <sup>ult</sup> ε <sub>x</sub> <sup>tu</sup> (µε)	Mean	4680		5860		6090		7250	
	S.Dev.	531		318		325		634	
	# of Specimens	90		7		9		5	
	# of Batches	21		4		9		1	
	Approval Class								
Poisson's Ratio ν <sub>xy</sub> <sup>t</sup> (MPa)	Mean								
	S.Dev.								
	# of Specimens								
	# of Batches								
	Approval Class								

Approval Class = F - Fully Approved, I - Interim, S - Screening

## Volume 5, Part D Data Requirements and Data Sets

<b>COMPOSITE</b>			
<i>Composite Name</i>		<b>Enhanced SiC/SiC</b>	<i>Manufacturer</i>
<i>Composite Description</i>		<b>SiC/ Carbon / SiC</b>	<i>Manuf. Lot #</i>
<i>Reinforc. Vol %</i>		<b>40%</b>	<i>Date of Manufacture</i>
<i>Density</i>		<b>2.3 g/cc</b>	<i>Fabrication Class</i>
<i>% Porosity</i>		<b>12%</b>	<i>Dev/Prod. Status</i>
			<b>Honeywell ACI</b>
			<b>multiple</b>
			<b>11/94 ... 12/97</b>
			<b>Chemical Vapor Infiltration</b>
			<b>Commercially Available</b>
<b>Reinforcement</b>	<i>Name</i>	<b>CG Nicalon™</b>	<i>Manufacturer</i>
<i>Composition</i>		<b>SiC</b>	<i>Structure/Architecture</i>
<i>Ply Count</i>		<b>11</b>	<i>Ply Thickness</i>
<i>Fabric Areal Weight</i>		<b>~287 g/m<sup>2</sup></b>	<i>Laminate Lay-up Sequence</i>
			<b>COI</b>
			<b>5-harness satin weave fabric</b>
			<b>~0.3 mm</b>
			<b>[0/90]<sub>ns</sub></b>
<b>Matrix</b>	<i>Composition</i>	<b>SiC</b>	<i>Source</i>
			<b>CVI</b>
<b>Interphase /Fiber Coating</b>			<i>Composition</i>
<i>Thickness</i>		<b>0.5µm</b>	<i>Source</i>
			<b>Carbon</b>
			<b>CVI</b>
<b>External Coating</b>	<i>Composition</i>	<b>SiC</b>	<i>Thickness</i>
<i>Fabrication Method</i>		<b>Chemical Vapor Infiltration</b>	<i>Source</i>
			<b>0.1 to 0.2 mm</b>
			<b>Chemical Vapor Infiltration</b>
<b>Compression Test Method</b>			
<i>Test Method Spec.</i>		<b>ASTM C 1358-96</b>	<i>Test File ID#</i>
<i>Modulus Calc Method</i>			<i>P.L. Calculation Method</i>
<i>Normalized by</i>			<i>Date of Testing</i>
			<b>multiple</b>
			<b>11/94 to 12/97</b>

Date of Data Submittal **Mar-01**

<b>Compression Test Data</b>		Temperature	<b>23</b>
		Atmosphere	<b>Air</b>
		Precondition	<b>None</b>
		Stress Orientation	<b>0°</b>
			<b>Normalized Measured</b>
Ultimate Compression Strength $F_x^{cu}$ (MPa)	Mean		<b>577</b>
	S.Dev.		<b>13.8</b>
	# of Specimens		<b>5</b>
	# of Batches		<b>5</b>
	Approval Class		
Elastic Modulus $E_x^c$ (MPa)	Mean		<b>141</b>
	S.Dev.		<b>11.7</b>
	# of Specimens		<b>5</b>
	# of Batches		<b>5</b>
	Approval Class		
Prop. Limit Stress $F_x^{pl}$ (MPa)	Mean		
	S.Dev.		
	# of Specimens		
	# of Batches		
	Approval Class		
Strain at $F_x^{cu}$ $\epsilon_x^{cu}$ (µε)	Mean		<b>0.433</b>
	S.Dev.		<b>0.055</b>
	# of Specimens		<b>5</b>
	# of Batches		<b>5</b>
	Approval Class		
Poisson's Ratio $\nu_{xy}^c$	Mean		
	S.Dev.		
	# of Specimens		
	# of Batches		
	Approval Class		

Approval Class = F - Fully Approved, I - Interim, S - Screening

Volume 5, Part D Data Requirements and Data Sets

<b>COMPOSITE</b>			
<i>Composite Name</i>	<b>Enhanced SiC/SiC</b>	<i>Manufacturer</i>	<b>Honeywell ACI</b>
<i>Composite Description</i>	<b>SiC/ Carbon / SiC</b>	<i>Manuf. Lot #</i>	<b>multiple</b>
<i>Reinforc. Vol %</i>	<b>40%</b>	<i>Date of Manufacture</i>	<b>11/94 ... 12/97</b>
<i>Density</i>	<b>2.3 g/cc</b>	<i>Fabrication Class</i>	<b>Chemical Vapor Infiltration</b>
<i>% Porosity</i>	<b>12%</b>	<i>Dev/Prod. Status</i>	<b>Commercially Available</b>
<b>Reinforcement</b>	<b>CG Nicalon™</b>	<i>Manufacturer</i>	<b>COI</b>
<i>Composition</i>	<b>SiC</b>	<i>Structure/Architecture</i>	<b>5-harness satin weave fabric</b>
<i>Ply Count</i>	<b>11</b>	<i>Ply Thickness</i>	<b>~0.3 mm</b>
<i>Fabric Areal Weight</i>	<b>~287 g/m<sup>2</sup></b>	<i>Laminate Lay-up Sequence</i>	<b>[0/90<sub>i</sub>]<sub>ns</sub></b>
<b>Matrix</b>	<b>SiC</b>	<i>Source</i>	<b>CVI</b>
<b>Interphase /Fiber Coating</b>		<i>Composition</i>	<b>Carbon</b>
<i>Thickness</i>	<b>0.5µm</b>	<i>Source</i>	<b>CVI</b>
<b>External Coating</b>	<b>SiC</b>	<i>Thickness</i>	<b>0.1 to 0.2 mm</b>
<i>Composition</i>	<b>Chemical Vapor Infiltration</b>	<i>Source</i>	<b>Chemical Vapor Infiltration</b>
<i>Fabrication Method</i>			
<b>Interlaminar Shear Test Method</b>		<i>Test File ID#</i>	<b>multiple</b>
<i>Test Method Spec.</i>	<b>ASTM C 1292-95a</b>	<i>Notch Distance</i>	<b>10 mm</b>
<i>Normalized by:</i>		<i>Date of Testing</i>	<b>11/94 to 12/97</b>

*Date of Data Submittal* **Mar-01**

<b>Shear Test Data</b>	<i>Temperature</i>	<b>23</b>	
	<i>Atmosphere</i>	<b>Air</b>	
	<i>Precondition</i>	<b>None</b>	
		<b>Normalized</b>	<b>Measured</b>
Ultimate In-Plane Shear Strength $F_{xy}^{su}$ (MPa)	<i>Mean</i>		<b>38.0</b>
	<i>S.Dev.</i>		<b>2.73</b>
	<i># of Specimens</i>		<b>2</b>
	<i># of Batches</i>		<b>1</b>
	<i>Approval Class</i>		

Approval Class = F - Fully Approved, I - Interim, S - Screening

Volume 5, Part D Data Requirements and Data Sets

<b>COMPOSITE</b>			
Composite Name	Enhanced SiC/SiC	Manufacturer	Honeywell ACI
Composite Description	SiC/ Carbon / SiC	Manuf. Lot #	multiple
Reinforc. Vol %	40%	Date of Manufacture	11/94 ... 12/97
Density	2.3 g/cc	Fabrication Class	Chemical Vapor Infiltration
% Porosity	12%	Dev/Prod. Status	Commercially Available
<b>Reinforcement</b> Name	CG Nicalon™	Manufacturer	COI
Composition	SiC	Structure/Architecture	5-harness satin weave fabric
Ply Count	11	Ply Thickness	~0.3 mm
Fabric Areal Weight	~287 g/m <sup>2</sup>	Laminate Lay-up Sequence	[0/90] <sub>ns</sub>
<b>Matrix</b> Composition	SiC	Source	CVI
<b>Interphase /Fiber Coating</b>		Composition	Carbon
Thickness	0.5µm	Source	CVI
<b>External Coating</b> Composition	SiC	Thickness	0.1 to 0.2 mm
Fabrication Method	Chemical Vapor Infiltration	Source	Chemical Vapor Infiltration
<b>Flexure Test Method</b>		Test File ID#	multiple
Test Method Spec.	C1161-94 & C1211-98a	Geometry (3 Pt/4 Pt)	4 Point
Outer Span Length( mm)	40 mm	Span/Depth Ratio	10 to 14
Modulus Calc Method	na	P.L. Calculation Method	na
Normalized by		Date of Testing	2/95 to 4/97

Date of Data Submittal **Mar-01**

		Normalized	Measured	Normalized	Measured
<b>Flexure Test Data</b>	Temperature	23		1200	
	Atmosphere	Air		Air	
	Precondition	None		None	
	Stress Orientation	0°		0°	
Ultimate Flexural Strength $F_x^{bu}$ (MPa)	Mean		420		447
	S.Dev.		36.6		30.9
	# of Specimens		27		20
	# of Batches		9		7
	Approval Class				
Elastic Modulus $E^b$ (GPa)	Mean				
	S.Dev.				
	# of Specimens				
	# of Batches				
	Approval Class				
Prop. Limit Stress $F^{pl}$ (MPa)	Mean				
	S.Dev.				
	# of Specimens				
	# of Batches				
	Approval Class				
Strain at $F^{bu}$ $\epsilon_x^{bu}$ (µε)	Mean				
	S.Dev.				
	# of Specimens				
	# of Batches				
	Approval Class				
Poisson's Ratio $\nu_{xy}^b$	Mean				
	S.Dev.				
	# of Specimens				
	# of Batches				
	Approval Class				

Approval Class = F - Fully Approved, I - Interim, S - Screening

18.2.3 CMC system #3

<b>COMPOSITE</b>			
<i>Composite Name</i>	<b>Carbon / SiC</b>	<i>Manufacturer</i>	<b>Honeywell ACI</b>
<i>Composite Description</i>	<b>Carbon / Carbon / CVI SiC</b>	<i>Manuf. Lot #</i>	<b>multiple</b>
<i>Reinforc. Vol %</i>	<b>45%</b>	<i>Date of Manufacture</i>	<b>3/91 to 2/99</b>
<i>Density</i>	<b>2.1 ±0.1 g/cc</b>	<i>Fabrication Class</i>	<b>Chemical Vapor Infiltration</b>
<i>% Porosity</i>	<b>~10%</b>	<i>Dev/Prod. Status</i>	<b>Commercially Available</b>
<b>Reinforcement</b>	<b>Carbon T300</b>	<i>Manufacturer</i>	<b>BP Amoco</b>
<i>Name</i>	<b>Carbon</b>	<i>Structure/Architecture</i>	<b>Plain weave fabric</b>
<i>Composition</i>	<b>27</b>	<i>Ply Thickness</i>	<b>~0.1 mm</b>
<i>Ply Count</i>	<b>~100 g/m<sup>2</sup></b>	<i>Laminate Lay-up Sequence</i>	<b>[0<sub>r</sub>/90<sub>i</sub>]</b>
<i>Fabric Areal Weight</i>			
<b>Matrix</b>	<b>SiC</b>	<i>Source</i>	<b>Chemical Vapor Infiltration</b>
<i>Composition</i>			
<b>Interphase /Fiber Coating</b>		<i>Composition</i>	<b>Carbon</b>
<i>Thickness</i>	<b>0.5µm</b>	<i>Source</i>	<b>Chemical Vapor Infiltration</b>
<b>External Coating</b>	<b>SiC</b>	<i>Thickness</i>	<b>0.1 to 0.2 mm</b>
<i>Composition</i>	<b>Chemical Vapor Infiltration</b>	<i>Source</i>	<b>Chemical Vapor Infiltration</b>
<i>Fabrication Method</i>			
<i>Date of Testing</i>	<b>3/91 TO 2/99</b>	<i>Date of Data Submittal</i>	<b>Mar-01</b>

<b>Summary Test Data</b>		<b>-157</b>	<b>23</b>	<b>600°C</b>	<b>1200°C</b>	<b>1400°C</b>
<i>Temperature</i>		<b>air</b>	<b>air</b>	<b>Inert</b>	<b>Inert</b>	<b>Inert</b>
<i>Atmosphere</i>		<b>none</b>	<b>none</b>	<b>none</b>	<b>none</b>	<b>none</b>
<i>Precondition</i>		<b>Measured</b>	<b>Measured</b>	<b>Measured</b>	<b>Measured</b>	<b>Measured</b>
<i>Normalized or Measured</i>						
Elastic Modulus	Mean	<b>57.9</b>	<b>70.2</b>	<b>102</b>	<b>114</b>	<b>95.7</b>
$E_x^t$ (GPa)	Approval Class	<b>S</b>	<b>S</b>	<b>S</b>	<b>S</b>	<b>S</b>
Elastic Modulus	Mean	<b>57.9</b>	<b>70.2</b>	<b>102</b>	<b>114</b>	<b>95.7</b>
$E_y^t$ (GPa)	Approval Class	<b>S</b>	<b>S</b>	<b>S</b>	<b>S</b>	<b>S</b>
Elastic Modulus	Mean					
$E_z^t$ (GPa)	Approval Class					
Poisson's Ratio $\nu_{xy}^t$	Mean					
	Approval Class					
Poisson's Ratio $\nu_{yz}^t$	Mean					
	Approval Class					
Poisson's Ratio $\nu_{zx}^t$	Mean					
	Approval Class					
Shear Modulus	Mean					
$G_{xy}^s$ (GPa)	Approval Class					
Shear Modulus	Mean					
$G_{yz}^s$ (MPa)	Approval Class					
Shear Modulus	Mean					
$G_{zx}^s$ (MPa)	Approval Class					
Prop. Limit Stress	Mean					
$F^{pl}$ MPa	Approval Class					
Thermal Conductivity	Mean					
$\alpha_x$	Approval Class					
Thermal Conductivity	Mean					
$\alpha_y$	Approval Class					
Thermal Conductivity	Mean					
$\alpha_z$	Approval Class					
Thermal Expansion	Mean					
$K_x$	Approval Class					
Thermal Expansion	Mean					
$K_y$	Approval Class					
Thermal Expansion	Mean					
$K_z$	Approval Class					

Approval Class = F - Fully Approved, I - Interim, S - Screening



<b>COMPOSITE</b>			
<i>Composite Name</i>	<b>Carbon / SiC</b>	<i>Manufacturer</i>	<b>Honeywell ACI</b>
<i>Composite Description</i>	<b>Carbon / Carbon / CVI SiC</b>	<i>Manuf. Lot #</i>	<b>multiple</b>
<i>Reinforc. Vol %</i>	<b>45%</b>	<i>Date of Manufacture</i>	<b>3/91 to 2/99</b>
<i>Density</i>	<b>2.1 ±0.1 g/cc</b>	<i>Fabrication Class</i>	<b>Chemical Vapor Infiltration</b>
<i>% Porosity</i>	<b>~10%</b>	<i>Dev/Prod. Status</i>	<b>Commercially Available</b>
<b>Reinforcement</b>	<b>Carbon T300</b>	<i>Manufacturer</i>	<b>BP Amoco</b>
<i>Name</i>	<b>Carbon</b>	<i>Structure/Architecture</i>	<b>Plain weave fabric</b>
<i>Composition</i>	<b>27</b>	<i>Ply Thickness</i>	<b>~0.1 mm</b>
<i>Ply Count</i>	<b>~100 g/m<sup>2</sup></b>	<i>Laminate Lay-up Sequence</i>	<b>[0/90;]</b>
<i>Fabric Areal Weight</i>			
<b>Matrix</b>	<b>SiC</b>	<i>Source</i>	<b>Chemical Vapor Infiltration</b>
<i>Composition</i>			
<b>Interphase /Fiber Coating</b>		<i>Composition</i>	<b>Carbon</b>
<i>Thickness</i>	<b>0.5µm</b>	<i>Source</i>	<b>Chemical Vapor Infiltration</b>
<b>External Coating</b>	<b>SiC</b>	<i>Thickness</i>	<b>0.1 to 0.2 mm</b>
<i>Composition</i>	<b>Chemical Vapor Infiltration</b>	<i>Source</i>	<b>Chemical Vapor Infiltration</b>
<i>Fabrication Method</i>			
<i>Date of Testing</i>	<b>3/91 TO 2/99</b>	<i>Date of Data Submittal</i>	<b>Mar-01</b>

<b>Summary Test Data</b>	Temperature	-157°C	23°C	600°C	1200°C	1400°C
		air	air	Inert	Inert	Inert
	Atmosphere	none	none	none	none	none
	Precondition	Measured	Measured	Measured	Measured	Measured
	Normalized or Measured	Measured	Measured	Measured	Measured	Measured
Tensile Strength $F_x^{tu}$ (MPa)	Mean	485	504	579	546	523
	Approval Class	S	S	S	S	S
Tensile Strength $F_y^{tu}$ (MPa)	Mean	485	504	579	546	523
	Approval Class	S	S	S	S	S
Tensile Strength $F_z^{tu}$ (MPa)	Mean					
	Approval Class					
Strain $\epsilon_x^{tu}$ (µε)	Mean	12300	10700	10500	11100	13800
	Approval Class	S	S	S	S	S
Strain $\epsilon_y^{tu}$ (µε)	Mean	12300	10700	10500	11100	13800
	Approval Class	S	S	S	S	S
Strain $\epsilon_z^{tu}$ (µε)	Mean					
	Approval Class					
Prop. Limit Stress $F_x^{pl}$ (MPa)	Mean					
	Approval Class					
Prop. Limit Stress $F_y^{pl}$ (MPa)	Mean					
	Approval Class					
Prop. Limit Stress $F_z^{pl}$ (MPa)	Mean					
	Approval Class					
Compressive Strength $F_x^{cu}$ (MPa)	Mean		545			
	Approval Class		S			
Compressive Strength $F_y^{cu}$ (MPa)	Mean		545			
	Approval Class		S			
Compressive Strength $F_z^{cu}$ (MPa)	Mean					
	Approval Class					
In-Plane Shear Strength $F_{xy}^{su}$ (MPa)	Mean		34.8			
	Approval Class		S			
Interlaminar Shear Strength $F_{yz}^{su}$ (MPa)	Mean		133			
	Approval Class		S			
Interlaminar Shear Strength $F_{zx}^{su}$ (MPa)	Mean		133			
	Approval Class		S			
Flexure Strength $F^b$ (MPa)	Mean					
	Approval Class					

Approval Class = F - Fully Approved, I - Interim, S - Screening

<b>COMPOSITE</b>		<b>Carbon / SiC</b>		<i>Manufacturer</i>		<b>Honeywell ACI</b>			
<i>Composite Name</i>		<b>Carbon / Carbon / CVI SiC</b>		<i>Manuf. Lot #</i>		<b>multiple</b>			
<i>Composite Description</i>		<b>45%</b>		<i>Date of Manufacture</i>		<b>3/91 to 2/99</b>			
<i>Reinforc. Vol %</i>		<b>2.1 ±0.1 g/cc</b>		<i>Fabrication Class</i>		<b>Chemical Vapor Infiltration</b>			
<i>Density</i>		<b>~10%</b>		<i>Dev/Prod. Status</i>		<b>Commercially Available</b>			
<i>% Porosity</i>									
<b>Reinforcement</b>		<b>Carbon T300</b>		<i>Manufacturer</i>		<b>BP Amoco</b>			
<i>Name</i>		<b>Carbon</b>		<i>Structure/Architecture</i>		<b>Plain weave fabric</b>			
<i>Composition</i>		<b>27</b>		<i>Ply Thickness</i>		<b>~0.1 mm</b>			
<i>Ply Count</i>		<b>100 g/m<sup>2</sup></b>		<i>Laminate Lay-up Sequence</i>		<b>[0/90]<sub>s</sub></b>			
<i>Fiber Areal Weight</i>									
<b>Matrix</b>		<b>SiC</b>		<i>Source</i>		<b>Chemical Vapor Infiltration</b>			
<i>Composition</i>				<i>Composition</i>		<b>Carbon</b>			
				<i>Source</i>		<b>Chemical Vapor Infiltration</b>			
<b>Interphase /Fiber Coating</b>		<b>0.5µm</b>							
<i>Thickness</i>									
<b>External Coating</b>		<b>SiC</b>		<i>Thickness</i>		<b>0.1 to 0.2 mm</b>			
<i>Composition</i>		<b>Chemical Vapor Infiltration</b>		<i>Source</i>		<b>Chemical Vapor Infiltration</b>			
<i>Fabrication Method</i>									
<b>Tensile Test Method</b>				<i>Test File ID#</i>		<b>multiple</b>			
<i>Test Method Spec.</i>		<b>C1275-95 &amp; C1359-96</b>		<i>Date of Testing</i>		<b>3/91 to 2/99</b>			
<i>Normalized by</i>				<i>Load/Ext. Strain Rate</i>		<b>0.5 mm/min.</b>			
<i>Modulus Calc Method</i>		<b>Instron Series IX FN. 19.3</b>		<i>P.L. Calculation Method</i>		<b>none</b>			
<i>Date of Data Submittal</i>						<b>Mar-01</b>			
<b>Tension Test</b>		<b>-157° C</b>		<b>23°C</b>		<b>600°C</b>		<b>1200°C</b>	
<i>Temperature</i>		<b>Air</b>		<b>Air</b>		<b>Inert</b>		<b>Inert</b>	
<i>Atmosphere</i>		<b>none</b>		<b>none</b>		<b>none</b>		<b>none</b>	
<i>Precondition</i>		<b>25.4 mm</b>		<b>25.4 mm</b>		<b>25.4 mm</b>		<b>25.4 mm</b>	
<i>Gage Length</i>		<b>0°</b>		<b>0°</b>		<b>0°</b>		<b>0°</b>	
<i>Stress</i>									
<i>Orientation</i>									
		<b>Normalized</b>		<b>Measured</b>		<b>Normalized</b>		<b>Measured</b>	
Ultimate Tensile Strength $F_x^{tu}$ (MPa)	Mean				<b>485</b>				<b>504</b>
	S.Dev.								<b>49.3</b>
	# of Specimens				<b>1</b>				<b>188</b>
	# of Batches				<b>1</b>				<b>31</b>
Elastic Modulus $F_y^{tu}$ (GPa)	Mean				<b>57.9</b>				<b>70.2</b>
	S.Dev.								<b>10.7</b>
	# of Specimens				<b>1</b>				<b>185</b>
	# of Batches				<b>1</b>				<b>30</b>
Prop. Limit Stress $F_x^{pl}$ (MPa)	Mean								
	S.Dev.								
	# of Specimens								
	# of Batches								
Strain at $F^{ut}$ $\epsilon_x^{tu}$ (µε)	Mean				<b>12300</b>				<b>10700</b>
	S.Dev.								<b>1790</b>
	# of Specimens				<b>1</b>				<b>188</b>
	# of Batches				<b>1</b>				<b>31</b>
Poisson's Ratio $\nu_{xy}^t$	Mean								
	S.Dev.								
	# of Specimens								
	# of Batches								
<i>Approval Class</i>									

Approval Class = F - Fully Approved, I - Interim, S - Screening

Volume 5, Part D Data Requirements and Data Sets

<b>COMPOSITE</b>			
Composite Name	Carbon / SiC	Manufacturer	Honeywell ACI
Composite Description	Carbon / Carbon / CVI SiC	Manuf. Lot #	multiple
Reinforc. Vol %	45%	Date of Manufacture	3/91 to 2/99
Density	2.1 ±0.1 g/cc	Fabrication Class	Chemical Vapor Infiltration
% Porosity	~10%	Dev/Prod. Status	Commercially Available
<b>Reinforcement</b>			
Name	Carbon T300	Manufacturer	BP Amoco
Composition	Carbon	Structure/Architecture	Plain weave fabric
Ply Count	27	Ply Thickness	~0.1 mm
Fiber Areal Weight	~100 g/m <sup>2</sup>	Laminate Lay-up Sequence	[0/90,]
<b>Matrix</b>			
Composition	SiC	Source	Chemical Vapor Infiltration
<b>Interphase /Fiber Coating</b>			
Thickness	0.5µm	Composition	Carbon
		Source	Chemical Vapor Infiltration
<b>External Coating</b>			
Composition	SiC	Thickness	0.1 to 0.2 mm
Fabrication Method	Chemical Vapor Infiltration	Source	Chemical Vapor Infiltration
<b>Tensile Test Method</b>			
Test Method Spec.	C1275-95 & C1359-96	Test File ID#	multiple
Normalized by		Date of Testing	3/91 to 2/99
Modulus Calc Method	Instron Series IX FN. 19.3	Load/Ext. Strain Rate	0.5 mm/min.
		P.L. Calculation Method	none

Date of Data Submittal

Mar-01

<b>Tension Test Data</b>	Temperature	1400°C	
	Atmosphere	Inert	
	Precondition	none	
	Gage Length	25.4 mm	
	Stress Orientation	0°	
		<b>Normalized</b>	<b>Measured</b>
Ultimate Tensile Strength $F_x^{tu}$ (MPa)	Mean		523
	S.Dev.		70.0
	# of Specimens		12
	# of Batches		11
	Approval Class		
Elastic Modulus $E_x^t$ (GPa)	Mean		95.7
	S.Dev.		24.4
	# of Specimens		12
	# of Batches		11
	Approval Class		
Prop. Limit Stress $F_x^{pl}$ (MPa)	Mean		
	S.Dev.		
	# of Specimens		
	# of Batches		
	Approval Class		
Strain at $F^{ut}$ $\epsilon_x^{tu}$ (µε)	Mean		13800
	S.Dev.		2800
	# of Specimens		10
	# of Batches		9
	Approval Class		
Poisson's Ratio $\nu_{xy}^t$	Mean		
	S.Dev.		
	# of Specimens		
	# of Batches		
	Approval Class		

Approval Class = F - Fully Approved, I - Interim, S - Screening

Volume 5, Part D Data Requirements and Data Sets

<b>COMPOSITE</b>			
<i>Composite Name</i>	<b>Carbon / SiC</b>	<i>Manufacturer</i>	<b>Honeywell ACI</b>
<i>Composite Description</i>	<b>Carbon / Carbon / CVI SiC</b>	<i>Manuf. Lot #</i>	<b>multiple</b>
<i>Reinforc. Vol %</i>	<b>45%</b>	<i>Date of Manufacture</i>	<b>3/91 to 2/99</b>
<i>Density</i>	<b>2.1 ±0.1 g/cc</b>	<i>Fabrication Class</i>	<b>Chemical Vapor Infiltration</b>
<i>% Porosity</i>	<b>~10%</b>	<i>Dev/Prod. Status</i>	<b>Commercially Available</b>
<b>Reinforcement</b> <i>Name</i>	<b>Carbon T300</b>	<i>Manufacturer</i>	<b>BP Amoco</b>
<i>Composition</i>	<b>Carbon</b>	<i>Structure/Architecture</i>	<b>Plain weave fabric</b>
<i>Ply Count</i>	<b>27</b>	<i>Ply Thickness</i>	<b>~0.1 mm</b>
<i>Fiber Areal Weight</i>	<b>~100 g/m<sup>2</sup></b>	<i>Laminate Lay-up Sequence</i>	<b>[0/90]<sub>s</sub></b>
<b>Matrix</b> <i>Composition</i>	<b>SiC</b>	<i>Source</i>	<b>Chemical Vapor Infiltration</b>
<b>Interphase /Fiber Coating</b>		<i>Composition</i>	<b>Carbon</b>
<i>Thickness</i>	<b>0.5µm</b>	<i>Source</i>	<b>Chemical Vapor Infiltration</b>
<b>External Coating</b>			
<i>Composition</i>	<b>SiC</b>	<i>Thickness</i>	<b>0.1 to 0.2 mm</b>
<i>Fabrication Method</i>	<b>Chemical Vapor Infiltration</b>	<i>Source</i>	<b>Chemical Vapor Infiltration</b>
<b>Tensile Test Method</b>		<i>Test File ID#</i>	<b>multiple</b>
<i>Test Method Spec.</i>	<b>C1275-95 &amp; C1359-96</b>	<i>Date of Testing</i>	<b>3/91 to 2/99</b>
<i>Normalized by</i>		<i>Load/Ext. Strain Rate</i>	<b>0.5 mm/min.</b>
<i>Modulus Calc Method</i>	<b>Instron Series IX FN. 19.3</b>	<i>P.L. Calculation Method</i>	<b>none</b>

**Date of Data Submittal**

**Mar-01**

<b>Compression Test Data</b>	<i>Temperature</i>	<b>23°C</b>	
	<i>Atmosphere</i>	<b>Air</b>	
	<i>Precondition</i>	<b>none</b>	
	<i>Gage Length</i>	<b>0°</b>	
	<i>Stress Orientation</i>	<b>23°C</b>	
		<b>Normalized</b>	<b>Measured</b>
Ultimate Compression Strength $F_x^{cu}$ (MPa)	Mean		<b>545</b>
	S.Dev.		<b>43</b>
	# of Specimens		<b>59</b>
	# of Batches		<b>21</b>
	Approval Class		
Elastic Modulus $E_x^c$ (MPa)	Mean		<b>101</b>
	S.Dev.		<b>7.14</b>
	# of Specimens		<b>56</b>
	# of Batches		<b>21</b>
	Approval Class		
Prop. Limit Stress $F_x^{pl}$ (MPa)	Mean		
	S.Dev.		
	# of Specimens		
	# of Batches		
	Approval Class		
Strain at $F_x^{cu}$ $\epsilon_x^{cu}$ (µε)	Mean		<b>3807</b>
	S.Dev.		<b>717</b>
	# of Specimens		<b>55</b>
	# of Batches		<b>21</b>
	Approval Class		
Poisson's Ratio $\nu_{xy}^c$	Mean		
	S.Dev.		
	# of Specimens		
	# of Batches		
	Approval Class		

Approval Class = F - Fully Approved, I - Interim, S - Screening

Volume 5, Part D Data Requirements and Data Sets

<b>COMPOSITE</b>			
Composite Name	Carbon / SiC	Manufacturer	Honeywell ACI
Composite Description	Carbon / Carbon / CVI SiC	Manuf. Lot #	multiple
Reinforc. Vol %	45%	Date of Manufacture	3/91 to 2/99
Density	2.1 ±0.1 g/cc	Fabrication Class	Chemical Vapor Infiltration
% Porosity	~10%	Dev/Prod. Status	Commercially Available
<b>Reinforcement</b>			
Name	Carbon T300	Manufacturer	BP Amoco
Composition	Carbon	Structure/Architecture	Plain weave fabric
Ply Count	27	Ply Thickness	~0.1 mm
Fabric Areal Weight	~100 g/m <sup>2</sup>	Laminate Lay-up Sequence	[0 <sub>i</sub> /90 <sub>i</sub> ]
<b>Matrix</b>			
Composition	SiC	Source	Chemical Vapor Infiltration
<b>Interphase /Fiber Coating</b>			
Thickness	0.5µm	Composition	Carbon
		Source	Chemical Vapor Infiltration
<b>External Coating</b>			
Composition	SiC	Thickness	0.1 to 0.2 mm
Fabrication Method	Chemical Vapor Infiltration	Source	Chemical Vapor Infiltration
<b>Tensile Test Method</b>			
Test Method Spec.	ASTM C 1292-95a	Test File ID#	multiple
Normalized by		Notch Distance	10 mm
		Date of Testing	3/91 to 2/99
Date of Data Submittal		Mar-01	

<b>Shear Test Data</b>	Temperature	-157°C		23°C	
	Atmosphere	Air		Air	
	Precondition	None		None	
		Normalized	Measured	Normalized	Measured
Ultimate In-Plane Strength F <sub>xy</sub> <sup>su</sup> (MPa)	Mean		22.8		34.8
	S.Dev.		na		6.47
	# of Specimens		1		267
	# of Batches		1		24
	Approval Class		S		F

<b>Interlaminar Shear Test Data</b>	Temperature	23	
	Atmosphere	Air	
	Precondition	none	
		Normalized	Measured
Ultimate Inter-laminar Strength F <sub>yz</sub> <sup>su</sup> (MPa)	Mean		133
	S.Dev.		na
	# of Specimens		1
	# of Batches		1
	Approval Class		S

Approval Class = F - Fully Approved, I - Interim, S - Screening

18.2.4 CMC system #4

<b>Composite</b>			
Composite Name	Hi-Nicalon/ Mi SiC	Manufacturer	Honeywell ACI
Composite Description	SiC / BN / Mi SiC	Manuf. Lot #	multiple
Reinforc. Vol %	34-40%	Date of Manufacture	2/98 ... 6/99
Density	2.6-2.75 g/cc	Fabrication Class	Chemical Vapor Infiltration
% Porosity	<5%	Dev/Prod. Status	Commercially Available
<b>Reinforcement</b> Name		Hi-Nicalon	Manufacturer
Composition		SiC	Structure/Architecture
Ply Count		8-12	Ply Thickness
Fiber Areal Weight		~285 g/m <sup>2</sup>	Laminate Lay-up Sequence
<b>Matrix</b> Composition		SiC + Siliconized SiC	Source
<b>Interphase /Fiber Coating</b>			Composition
Thickness		-0.5µm	Source
<b>External Coating</b> Composition		None	Thickness
Fabrication Method		na	Source
Date of Testing		2/98 to 6/99	Date of Data Submittal
			Mar-01
<b>Summary Test Data</b>		Temperature	23°C
		Atmosphere	1204°C
		Precondition	Air
			Air
		Normalized or Measured	none
			none
			Measured
Elastic Modulus E <sub>x</sub> <sup>t</sup> (GPa)	Mean	196	144
	Approval Class	S	S
Elastic Modulus E <sub>y</sub> <sup>t</sup> (GPa)	Mean	196	144
	Approval Class	S	S
Elastic Modulus E <sub>z</sub> <sup>t</sup> (GPa)	Mean		
	Approval Class		
Poisson's Ratio ν <sub>xy</sub> <sup>t</sup>	Mean		
	Approval Class		
Poisson's Ratio ν <sub>yz</sub> <sup>t</sup>	Mean		
	Approval Class		
Poisson's Ratio ν <sub>zx</sub> <sup>t</sup>	Mean		
	Approval Class		
Shear Modulus G <sub>xy</sub> (GPa)	Mean		
	Approval Class		
Shear Modulus G <sub>yz</sub> (MPa)	Mean		
	Approval Class		
Shear Modulus G <sub>zx</sub> (MPa)	Mean		
	Approval Class		
Prop. Limit Stress F <sup>pl</sup> (MPa)	Mean		
	Approval Class		
Thermal Conductivity α <sub>x</sub>	Mean		
	Approval Class		
Thermal Conductivity α <sub>y</sub>	Mean		
	Approval Class		
Thermal Conductivity α <sub>z</sub>	Mean		
	Approval Class		
Thermal Expansion K <sub>x</sub>	Mean		
	Approval Class		
Thermal Expansion K <sub>y</sub>	Mean		
	Approval Class		
Thermal Expansion K <sub>z</sub>	Mean		
	Approval Class		

Approval Class = F - Fully Approved, I - Interim, S - Screening

<b>Composite</b>			
Composite Name	Hi-Nicalon/ Mi SiC	Manufacturer	Honeywell ACI
Composite Description	SiC / BN / Mi SiC	Manuf. Lot #	multiple
Reinforc. Vol %	34-40%	Date of Manufacture	2/98 ... 6/99
Density	2.6-2.75 g/cc	Fabrication Class	Chemical Vapor Infiltration
% Porosity	<5%	Dev/Prod. Status	Commercially Available
<b>Reinforcement</b>	Name	Hi-Nicalon	Manufacturer
Composition		SiC	Structure/Architecture
Ply Count		8-12	Ply Thickness
Fiber Areal Weight		~285 g/m <sup>2</sup>	Laminate Lay-up Sequence
			COI
			5-harness satin weave fabric
			~0.3 mm
			[0/90] <sub>s</sub>
<b>Matrix</b>	Composition	SiC + Siliconized SiC	Source
			Slurry Cast Melt Infiltration
<b>Interphase /Fiber Coating</b>		Composition	Boron Nitride
Thickness		~0.5µm	Source
			Chemical Vapor Infiltration
<b>External Coating</b>	Composition	None	Thickness
Fabrication Method		na	Source
			na
			na
Date of Testing	2/98 to 6/99	Date of Data Submittal	Mar-01
<b>Summary</b>	Temperature	23°C	1204°C
	Atmosphere	Air	Air
	Precondition	none	none
	Normalized or Measured	Measured	Measured
Tensile Strength $F_x^{tu}$ (MPa)	Mean	358	271
	Approval Class	S	S
Tensile Strength $F_y^{tu}$ (MPa)	Mean		
	Approval Class		
Tensile Strength $F_z^{tu}$ (MPa)	Mean		
	Approval Class		
Strain $\epsilon_x^{tu}$ (µε)	Mean	7430	5190
	Approval Class	S	S
Strain $\epsilon_y^{tu}$ (µε)	Mean	7430	5190
	Approval Class	S	S
Strain $\epsilon_z^{tu}$ (µε)	Mean		
	Approval Class		
Prop. Limit Stress $F_x^{pl}$ (MPa)	Mean	120	130
	Approval Class	S	S
Prop. Limit Stress $F_y^{pl}$ (MPa)	Mean	120	130
	Approval Class	S	S
Prop. Limit Stress $F_z^{pl}$ (MPa)	Mean		
	Approval Class		
Compressive Strength $F_x^{cu}$ (MPa)	Mean		
	Approval Class		
Compressive Strength $F_y^{cu}$ (MPa)	Mean		
	Approval Class		
Compressive Strength $F_z^{cu}$ (MPa)	Mean		
	Approval Class		
In-Plane Shear Strength $F_{xy}^{su}$ (MPa)	Mean		
	Approval Class		
Interlaminar Shear Strength $F_{yz}^{su}$ (MPa)	Mean		
	Approval Class		
Interlaminar Shear Strength $F_{zx}^{su}$ (MPa)	Mean		
	Approval Class		
Flexure Strength $F^{bu}$ (MPa)	Mean		
	Approval Class		

Approval Class = F - Fully Approved, I - Interim, S - Screening

<b>COMPOSITE</b>			
Composite Name	Hi-Nicalon/ Mi SiC	Manufacturer	Honeywell ACI
Composite Description	SiC / BN / Mi SiC	Manuf. Lot #	multiple
Reinforc. Vol %	34-40%	Date of Manufacture	2/98 ... 6/99
Density	2.6-2.75 g/cc	Fabrication Class	Chemical Vapor Infiltration
% Porosity	<5%	Dev/Prod. Status	Commercially Available
<b>Reinforcement</b> Name	Hi-Nicalon	Manufacturer	COI
Composition	SiC	Structure/Architecture	5-harness satin weave fabric
Ply Count	8-12	Ply Thickness	~0.3 mm
Fiber Areal Weight	~285 g/m <sup>2</sup>	Laminate Lay-up Sequence	[0 <sub>i</sub> /90 <sub>i</sub> ] <sub>n</sub>
<b>Matrix</b> Composition	SiC + Siliconized SiC	Source	Slurry Melt Cast Infiltration
<b>Interphase /Fiber Coating</b>		Composition	Boron Nitride
Thickness	na	Source	Chemical Vapor Infiltration
<b>External Coating</b>	~0.5µm		na
Composition	None	Thickness	na
Fabrication Method	na	Source	na
<b>Tensile Test Method</b>		Test File ID#	multiple
Test Method Spec.	C1275-95 & C1359-96	Date of Testing	2/98 to 6/99
Normalized by:		Load/Ext. Strain Rate	0.5 mm / min.
Modulus Calc Method	from 3.5 to 69 MPa LS fit	P.L. Calculation Method	Offset Yield (0.005%)

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<b>Tension Test Data</b>	Temperature	23	23		
	Atmosphere	Air	Air		
	Precondition	none	none		
	Gage Length	na	na		
	Stress Orientation	0°	0°		
			Normalized	Measured	Normalized
Ultimate Tensile Strength $F_x^{tu}$ (MPa)	Mean	358		271	
	S.Dev.	34.6		19.6	
	# of Specimens	19		3	
	# of Batches	10		2	
	Approval Class	S		S	
Elastic Modulus $E_x^t$ (GPa)	Mean	196		145	
	S.Dev.	15.2		5.79	
	# of Specimens	19		3	
	# of Batches	10		2	
	Approval Class	S		S	
Prop. Limit Stress $F_x^{pl}$ (MPa)	Mean	120		130	
	S.Dev.	10.8		22.3	
	# of Specimens	19		3	
	# of Batches	10		2	
	Approval Class	S		S	
Strain at $F^{ut}$ $\epsilon_x^{tu}$ (µε)	Mean	7430		5190	
	S.Dev.	1050		480	
	# of Specimens	19		3	
	# of Batches	10		2	
	Approval Class	S		S	
Poisson's Ratio $\nu_{xy}^t$	Mean				
	S.Dev.				
	# of Specimens				
	# of Batches				
	Approval Class				

Approval Class = F - Fully Approved, I - Interim, S - Screening



18.2.5 CMC system #5

<b>Composite</b>				
Composite Name		AS-N720-1	Manufacturer	COI Ceramics
Composite Description		Oxide / Oxide		Various
Reinforc. Vol %		45	Date of Manufacture	1997 to 1999
Density		2.54 g/cc	Fabrication Class	Sol-Gel
% Porosity		25.5	Dev/Prod. Status	Commercially Available
<b>Reinforcement</b>	Name	Nextel 720	Manufacturer	3M
Composition		Alumina and Mullite	Structure/Architecture	8-harness satin weave fabric
Ply Count		12	Ply Thickness	0.229 mm
Fiber Areal Weight			Laminate Lay-up Sequence	[0/90] <sub>3s</sub>
<b>Matrix</b>	Composition	Alumino-silicate	Source	Sol-gel
<b>Interphase /Fiber Coating</b>			Composition	none
Thickness		na	Source	na
<b>External Coating</b>	Composition	none	Thickness	na
Fabrication Method		na	Source	na
	Date of Testing	na	Date of Data Submittal	Mar-01
<b>Summary Test Data</b>				
	Temperature	23		
	Atmosphere	Air		
	Precondition	None		
	Normalized or Measured	Measured		
Elastic Modulus $E_x^t$ (GPa)	Mean	75.8		
	Approval Class			
Elastic Modulus $E_y^t$ (GPa)	Mean			
	Approval Class			
Elastic Modulus $E_z^t$ (GPa)	Mean			
	Approval Class			
Poisson's Ratio $\nu_{xy}^t$	Mean			
	Approval Class			
Poisson's Ratio $\nu_{yz}^t$	Mean			
	Approval Class			
Poisson's Ratio $\nu_{zx}^t$	Mean			
	Approval Class			
Shear Modulus $G_{xy}^s$ (GPa)	Mean			
	Approval Class			
Shear Modulus $G_{yz}^s$ (MPa)	Mean			
	Approval Class			
Shear Modulus $G_{zx}^s$ (MPa)	Mean			
	Approval Class			
Prop. Limit Stress $F^{pl}$ MPa	Mean			
	Approval Class			
Thermal Conductivity $\alpha_x$	Mean			
	Approval Class			
Thermal Conductivity $\alpha_y$	Mean			
	Approval Class			
Thermal Conductivity $\alpha_z$	Mean			
	Approval Class			
Thermal Expansion $K_x$	Mean			
	Approval Class			
Thermal Expansion $K_y$	Mean			
	Approval Class			
Thermal Expansion $K_z$	Mean			
	Approval Class			

Approval Class = F - Fully Approved, I - Interim, S - Screening

## Volume 5, Part D Data Requirements and Data Sets

<b>Composite</b>			
<i>Composite Name</i>		<b>AS-N720-1</b>	<i>Manufacturer</i>
<i>Composite Description</i>		<b>Oxide / Oxide</b>	<i>Manuf. Lot #</i>
<i>Reinforc. Vol %</i>		<b>45</b>	<i>Date of Manufacture</i>
<i>Density</i>		<b>2.54 g/cc</b>	<i>Fabrication Class</i>
<i>% Porosity</i>		<b>25.5</b>	<i>Dev/Prod. Status</i>
			<b>COI Ceramics</b>
			<b>Various</b>
			<b>1997 to 1999</b>
			<b>Sol-Gel</b>
			<b>Commercially Available</b>
<b>Reinforcement</b>		<b>Nextel 720</b>	<i>Manufacturer</i>
<i>Name</i>		<b>Alumina and Mullite</b>	<i>Structure/Architecture</i>
<i>Composition</i>		<b>12</b>	<i>Ply Thickness</i>
<i>Ply Count</i>			<i>Laminate Lay-up Sequence</i>
<i>Fiber Areal Weight</i>			<b>[0/90<sub>i</sub>]<sub>3s</sub></b>
<b>Matrix</b>		Alumino-silicate	<i>Source</i>
<i>Composition</i>			<b>Sol-gel</b>
<b>Interphase /Fiber Coating</b>			<i>Composition</i>
<i>Thickness</i>		<b>na</b>	<i>Source</i>
<b>External Coating</b>		<b>none</b>	<i>Thickness</i>
<i>Fabrication Method</i>		<b>na</b>	<i>Source</i>
			<b>na</b>
<i>Date of Testing</i>		<b>na</b>	<i>Date of Data Submittal</i>
			<b>Mar-01</b>
<b>Summary Test Data</b>		<b>23</b>	
<i>Temperature</i>		<b>Air</b>	
<i>Atmosphere</i>		<b>None</b>	
<i>Precondition</i>		<b>Measured</b>	
<i>Normalized or Measured</i>			
Tensile Strength $F_x^{tu}$ (MPa)	Mean	<b>222</b>	
	Approval Class		
Tensile Strength $F_y^{tu}$ (MPa)	Mean		
	Approval Class		
Tensile Strength $F_z^{tu}$ (MPa)	Mean		
	Approval Class		
Strain $\epsilon_x^{tu}$ ( $\mu\epsilon$ )	Mean	<b>3670</b>	
	Approval Class		
Strain $\epsilon_y^{tu}$ ( $\mu\epsilon$ )	Mean		
	Approval Class		
Strain $\epsilon_z^{tu}$ ( $\mu\epsilon$ )	Mean		
	Approval Class		
Prop. Limit Stress $F_x^{pl}$ (MPa)	Mean		
	Approval Class		
Prop. Limit Stress $F_y^{pl}$ (MPa)	Mean		
	Approval Class		
Prop. Limit Stress $F_z^{pl}$ (MPa)	Mean		
	Approval Class		
Compression Strength $F_x^{cu}$ (MPa)	Mean	<b>239</b>	
	Approval Class		
Compression Strength $F_y^{cu}$ (MPa)	Mean		
	Approval Class		
Compression Strength $F_z^{cu}$ (MPa)	Mean		
	Approval Class		
In-Plane Shear Strength $F_{xy}^{su}$ (MPa)	Mean	<b>14.4</b>	
	Approval Class		
Interlaminar Shear Strength $F_{yz}^{su}$ (MPa)	Mean	<b>43.4</b>	
	Approval Class		
Interlaminar Shear Strength $F_{zx}^{su}$ (MPa)	Mean		
	Approval Class		
Flexure Strength $F^b$ (MPa)	Mean		
	Approval Class		

Approval Class = F - Fully Approved, I - Interim, S - Screening

Volume 5, Part D Data Requirements and Data Sets

<b>COMPOSITE</b>			
Composite Name	AS-N720-1	Manufacturer	COI Ceramics
Composite Description	Oxide / Oxide	Manuf. Lot #	Various
Reinforc. Vol %	45	Date of Manufacture	1997 to 1999
Density	2.54 g/cc	Fabrication Class	Sol-Gel
% Porosity	25.5	Dev/Prod. Status	Commercially Available
<b>Reinforcement</b> Name	Nextel 720	Manufacturer	3M
Composition	Alumina and Mullite	Structure/Architecture	8-harness satin weave fabric
Ply Count	12	Ply Thickness	0.229 mm
Fiber Areal Weight		Laminate Lay-up Sequence	[0/90 <sub>r</sub> ] <sub>3s</sub>
<b>Matrix</b> Composition	Alumino-silicate	Source	Sol-gel
<b>Interphase /Fiber Coating</b>		Composition	none
Thickness	na	Source	na
<b>External Coating</b>			
Composition	none	Thickness	na
Fabrication Method	na	Source	na
<b>Tensile Test Method</b>		Test File ID#	na
Test Method Spec.	ASTM D 3039	Date of Testing	na
Normalized by:	Not normalized	Load/Ext. Strain Rate	0.05 in/min
Modulus Calc Method	Secant method from 0.01% to 0.05% strain	P.L. Calculation Method	none

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<b>Tension Test Data</b>	Temperature	23	
	Atmosphere	Air	
	Precondition	none	
	Gage Length	na	
	Stress Orientation	0°	
		<b>Normalized</b>	<b>Measured</b>
Ultimate Tensile Strength $F_x^{tu}$ (MPa)	Mean		222
	S.Dev.		12.1
	# of Specimens		11
	# of Batches		na
	Approval Class		
Elastic Modulus $E_x^t$ (GPa)	Mean		75.8
	S.Dev.		1.1
	# of Specimens		8
	# of Batches		na
	Approval Class		
Prop. Limit Stress $F_x^{pl}$ (MPa)	Mean		
	S.Dev.		
	# of Specimens		
	# of Batches		
	Approval Class		
Strain at $F^{ut}$ $\epsilon_x^{tu}$ (µε)	Mean		3670
	S.Dev.		180
	# of Specimens		8
	# of Batches		na
	Approval Class		
Poisson's Ratio $\nu_{xy}^t$	Mean		
	S.Dev.		
	# of Specimens		
	# of Batches		
	Approval Class		

Approval Class = F - Fully Approved, I - Interim, S - Screening

Volume 5, Part D Data Requirements and Data Sets

<b>COMPOSITE</b>			
Composite Name	AS-N720-1	Manufacturer	COI Ceramics
Composite Description	Oxide / Oxide	Manuf. Lot #	Various
Reinforc. Vol %	45	Date of Manufacture	1997 to 1999
Density	2.54 g/cc	Fabrication Class	Sol-Gel
% Porosity	25.5	Dev/Prod. Status	Commercially Available
<b>Reinforcement</b>	<b>Nextel 720</b>	Manufacturer	<b>3M</b>
Name	Alumina and Mullite	Structure/Architecture	8-harness satin weave fabric
Composition		Ply Thickness	0.229 mm
Ply Count	12	Laminate Lay-up Sequence	[0/90 <sub>r</sub> ] <sub>3s</sub>
Fiber Areal Weight			
<b>Matrix</b>	<b>Alumino-silicate</b>	Source	<b>Sol-gel</b>
Composition			
<b>Interphase /Fiber Coating</b>		Composition	<b>none</b>
Thickness	na	Source	na
<b>External Coating</b>			
Composition	none	Thickness	na
Fabrication Method	na	Source	na
<b>Tensile Test Method</b>		Test File ID#	<b>na</b>
Test Method Spec.	ASTM D 3410	Date of Testing	na
Normalized by	na	Load/Ext. Strain Rate	0.02 in/min
Modulus Calc Method	Secant method from 0.01% to 0.05% strain	P.L. Calculation Method	none
<b>Date of Data Submittal</b>		<b>Mar-01</b>	

<b>Compression Test Data</b>		Temperature	23
		Atmosphere	Air
		Precondition	none
		Gage Length	na
		Stress Orientation	0°
		<b>Normalized</b>	<b>Measured</b>
Ultimate Compression Strength F <sub>x</sub> <sup>cu</sup> (MPa)	Mean		239
	S.Dev.		14.1
	# of Specimens		6
	# of Batches		1
	Approval Class		
Elastic Modulus E <sub>x</sub> <sup>c</sup> (MPa)	Mean		80.6
	S.Dev.		1.65
	# of Specimens		6
	# of Batches		1
	Approval Class		
Prop. Limit Stress F <sub>x</sub> <sup>pl</sup> (MPa)	Mean		
	S.Dev.		
	# of Specimens		
	# of Batches		
	Approval Class		
Strain at F <sup>cu</sup> ε <sub>x</sub> <sup>cu</sup> (μE)	Mean		2990
	S.Dev.		250
	# of Specimens		5
	# of Batches		na
	Approval Class		
Poisson's Ratio ν <sub>xy</sub> <sup>c</sup>	Mean		
	S.Dev.		
	# of Specimens		
	# of Batches		
	Approval Class		

Approval Class = F - Fully Approved, I - Interim, S - Screening

Volume 5, Part D Data Requirements and Data Sets

<b>COMPOSITE</b>			
Composite Name	AS-N720-1	Manufacturer	COI Ceramics
Composite Description	Oxide / Oxide	Manuf. Lot #	Various
Reinforc. Vol %	45	Date of Manufacture	1997 to 1999
Density	2.54 g/cc	Fabrication Class	Sol-Gel
% Porosity	25.5	Dev/Prod. Status	Commercially Available
<b>Reinforcement</b> Name	Nextel 720	Manufacturer	3M
Composition	Alumina and Mullite	Structure/Architecture	8 harness satin weave fabric
Ply Count	12	Ply Thickness	0.229 mm
Fiber Areal Weight		Laminate Lay-up Sequence	[0/90] <sub>i3s</sub>
<b>Matrix</b> Composition	Alumino-silicate	Source	Sol-gel
<b>Interphase /Fiber Coating</b>			
Thickness	n/a	Composition	none
		Source	n/a
<b>External Coating</b>			
Composition	n/a	Thickness	n/a
Fabrication Method	n/a	Source	n/a
<b>Interlaminar Shear Test Method</b>		Test File ID#	na
Test Method Spec.	na	Notch Distance	na
Normalized by:	na	Date of Testing	na
Date of Data Submittal		Mar-01	

<b>Shear Test Data</b>	Temperature	23	
	Atmosphere	Air	
	Precondition	none	
		<b>Normalized</b>	<b>Measured</b>
Ultimate In-Plane Strength $F_{xy}^{su}$ (MPa)	Mean		14.4
	S.Dev.		2.28
	# of Specimens		27
	# of Batches		na
	Approval Class		

<b>Interlaminar Shear Test Data</b>	Temperature	23	
	Atmosphere	Air	
	Precondition	none	
		<b>Normalized</b>	<b>Measured</b>
Ultimate Inter-laminar Strength $F_{yz}^{su}$ (MPa)	Mean		43.4
	S.Dev.		0.83
	# of Specimens		6
	# of Batches		1
	Approval Class		

Approval Class = F - Fully Approved, I - Interim, S - Screening

Volume 5, Part D Data Requirements and Data Sets

<b>COMPOSITE</b>			
Composite Name	AS-N720-1	Manufacturer	COI Ceramics
Composite Description	Oxide / Oxide	Manuf. Lot #	Various
Reinforc. Vol %	45	Date of Manufacture	1997 to 1999
Density	2.54 g/cc	Fabrication Class	Sol-Gel
% Porosity	25.5	Dev/Prod. Status	Commercially Available
<b>Reinforcement</b>	<b>Nextel 720</b>	Manufacturer	3M
Composition	Alumina and Mullite	Structure/Architecture	8-harness satin weave fabric
Ply Count	12	Ply Thickness	0.229 mm
Fiber Areal Weight		Laminate Lay-up Sequence	[0/90] <sub>3s</sub>
<b>Matrix</b>	<b>Alumino-silicate</b>	Source	Sol-gel
<b>Interphase /Fiber Coating</b>		Composition	none
Thickness	na	Source	na
<b>External Coating</b>			
Composition	none	Thickness	na
Fabrication Method	na	Source	na
<b>Flexure Test Method</b>		Test File ID#	na
Test Method Spec.	ASTM D 790	Geometry (3 Pt/4 Pt)	4 pt - 1/4 pt
Outer Span Length(mm)	122	Span/Depth Ratio	20
Modulus Calc Method	Secant method @ 0.07% and 0.05% strain	P.L. Calculation Method	none
Normalized by		Date of Testing	na

Date of Data Submittal

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<b>Flexure Test Data</b>	Temperature	23	
	Atmosphere	Air	
	Precondition	none	
	Stress Orientation	0°	
		Normalized	Measured
Ultimate Flexural Strength $F_x^{bu}$ (MPa)	Mean		259
	S.Dev.		40.4
	# of Specimens		27
	# of Batches		na
	Approval Class		
Elastic Modulus $E_x^b$ (GPa)	Mean		93.7
	S.Dev.		10.0
	# of Specimens		27
	# of Batches		na
	Approval Class		
Prop. Limit Stress $F_x^{pl}$ (MPa)	Mean		
	S.Dev.		
	# of Specimens		
	# of Batches		
	Approval Class		
Strain at $F_x^{bu}$ $\epsilon_x^{bu}$ (µε)	Mean		2770
	S.Dev.		370
	# of Specimens		27
	# of Batches		na
	Approval Class		
Poisson's Ratio $\nu_{xy}^b$	Mean		
	S.Dev.		
	# of Specimens		
	# of Batches		
	Approval Class		

Approval Class = F - Fully Approved, I - Interim, S - Screening

18.2.6 CMC system #6

<b>COMPOSITE</b>			
<i>Composite Name</i>		<b>Sylramic S-200</b>	<i>Manufacturer</i>
<i>Composite Description</i>		<b>SiC / BN / Si3N4</b>	<i>Manuf. Lot #</i>
<i>Reinforc. Vol %</i>		<b>45%</b>	<i>Date of Manufacture</i>
<i>Density</i>		<b>2.2 g/cc</b>	<i>Fabrication Class</i>
<i>% Porosity</i>		<b>2.7% Open</b>	<i>Dev/Prod. Status</i>
			<b>COI</b>
			<b>#14416</b>
			<b>Summer 1997</b>
			<b>Preceramic polymer</b>
			<b>Commercially Available</b>
<b>Reinforcement</b>	<i>Name</i>	<b>CG Nicalon™</b>	<i>Manufacturer</i>
<i>Composition</i>		<b>SiC</b>	<i>Structure/Architecture</i>
<i>Ply Count</i>		<b>8</b>	<i>Ply Thickness</i>
<i>Fabric Areal Weight</i>			<i>Laminate Lay-up Sequence</i>
			<b>[0/90/0/90/90/0/90/0]s</b>
<b>Matrix</b>	<i>Composition</i>	<b>Si-C-N w/Si3N4 Particle Filler</b>	<i>Source</i>
			<b>COI</b>
<b>Interphase /Fiber Coating</b>			<i>Composition</i>
<i>Thickness</i>		<b>0.5µm</b>	<i>Source</i>
			<b>Boron Nitride</b>
			<b>CVD</b>
<b>External Coating</b>	<i>Composition</i>	<b>None</b>	<i>Thickness</i>
<i>Fabrication Method</i>		<b>na</b>	<i>Source</i>
			<b>na</b>
<i>Date of Testing</i>	<b>Dec 97 to Oct 98</b>		<i>Date of Data Submittal</i>
			<b>Mar-01</b>

<b>Summary Test Data</b>	Temperature	<b>23</b>
	Atmosphere	<b>Air</b>
	Precondition	<b>none</b>
	Normalized or Measured	<b>Measured</b>
Elastic Modulus $E_x^t$ (GPa)	Mean	<b>93.0</b>
	Approval Class	
Elastic Modulus $E_y^t$ (GPa)	Mean	<b>93.0</b>
	Approval Class	
Elastic Modulus $E_z^t$ (GPa)	Mean	
	Approval Class	
Poisson's Ratio $\nu_{xy}^t$	Mean	
	Approval Class	
Poisson's Ratio $\nu_{yz}^t$	Mean	
	Approval Class	
Poisson's Ratio $\nu_{zx}^t$	Mean	
	Approval Class	
Shear Modulus $G_{xy}^s$ (GPa)	Mean	
	Approval Class	
Shear Modulus $G_{yz}^s$ (MPa)	Mean	
	Approval Class	
Shear Modulus $G_{zx}^s$ (MPa)	Mean	
	Approval Class	
Prop. Limit Stress $F^{Pl}$ MPa	Mean	
	Approval Class	
Thermal Conductivity $\alpha_x$	Mean	
	Approval Class	
Thermal Conductivity $\alpha_y$	Mean	
	Approval Class	
Thermal Conductivity $\alpha_z$	Mean	
	Approval Class	
Thermal Expansion $K_x$	Mean	
	Approval Class	
Thermal Expansion $K_y$	Mean	
	Approval Class	
Thermal Expansion $K_z$	Mean	
	Approval Class	

Approval Class = F - Fully Approved, I - Interim, S - Screening

<b>COMPOSITE</b>			
Composite Name	<b>Sylramic S-200</b>	Manufacturer	<b>COI</b>
Composite Description	<b>SiC / BN / Si3N4</b>	Manuf. Lot #	<b>#14416</b>
Reinforc. Vol %	<b>45%</b>	Date of Manufacture	<b>Summer 1997</b>
Density	<b>2.2 g/cc</b>	Fabrication Class	<b>Pre ceramic polymer</b>
% Porosity	<b>2.7% Open</b>	Dev/Prod. Status	<b>Commercially Available</b>
<b>Reinforcement</b>	<b>CG Nicalon™</b>	Manufacturer	<b>COI</b>
Composition	<b>SiC</b>	Structure/Architecture	<b>8 harness satin weave fabric</b>
Ply Count	<b>8</b>	Ply Thickness	<b>.346 mm</b>
Fabric Areal Weight		Laminate Lay-up Sequence	<b>0/90/0/0/90/90/0/90/0/</b>
<b>Matrix</b>	<b>Si-C-N w/Si3N4 Particle Filler</b>	Source	<b>COI</b>
<b>Interphase /Fiber Coating</b>		Composition	<b>Boron Nitride</b>
Thickness	<b>0.5µm</b>	Source	<b>CVD</b>
<b>External Coating</b>	<b>None</b>	Thickness	<b>na</b>
Fabrication Method	<b>na</b>	Source	<b>na</b>
Date of Testing	<b>Dec 97 to Oct 98</b>	Date of Data Submittal	<b>Mar-01</b>

<b>Summary Test Data</b>		Temperature	<b>23</b>
		Atmosphere	<b>Air</b>
		Precondition	<b>none</b>
		Normalized or Measured	<b>Measured</b>
Tensile Strength $F_x^{tu}$ (MPa)	Mean		<b>251</b>
	Approval Class		
Tensile Strength $F_y^{tu}$ (MPa)	Mean		<b>251</b>
	Approval Class		
Tensile Strength $F_z^{tu}$ (MPa)	Mean		
	Approval Class		
Strain $\epsilon_x^{tu}$ (µε)	Mean		<b>4310</b>
	Approval Class		
Strain $\epsilon_y^{tu}$ (µε)	Mean		<b>4310</b>
	Approval Class		
Strain $\epsilon_z^{tu}$ (µε)	Mean		
	Approval Class		
Prop. Limit Stress $F_x^{pl}$ (MPa)	Mean		<b>85.0</b>
	Approval Class		
Prop. Limit Stress $F_y^{pl}$ (MPa)	Mean		<b>85.0</b>
	Approval Class		
Prop. Limit Stress $F_z^{pl}$ (MPa)	Mean		
	Approval Class		
Compressive Strength $F_x^{cu}$ (MPa)	Mean		
	Approval Class		
Compressive Strength $F_y^{cu}$ (MPa)	Mean		
	Approval Class		
Compressive Strength $F_z^{cu}$ (MPa)	Mean		
	Approval Class		
In-Plane Shear Strength $F_{xy}^{su}$ (MPa)	Mean		<b>33.0</b>
	Approval Class		
Interlaminar Shear Strength $F_{yz}^{su}$ (MPa)	Mean		<b>111</b>
	Approval Class		
Interlaminar Shear Strength $F_{zx}^{su}$ (MPa)	Mean		<b>111</b>
	Approval Class		
Flexure Strength $F^b$ (MPa)	Mean		<b>339</b>
	Approval Class		

Approval Class = F - Fully Approved, I - Interim, S - Screening



Volume 5, Part D Data Requirements and Data Sets

<b>COMPOSITE</b>			
Composite Name	Sylramic S-200	Manufacturer	COI
Composite Description	SiC / BN / Si3N4	Manuf. Lot #	#14416
Reinforc. Vol %	45%	Date of Manufacture	Summer 1997
Density	2.2 g/cc	Fabrication Class	Preceramic polymer
% Porosity	2.7% Open	Dev/Prod. Status	Commercially Available
<b>Reinforcement</b>			
Composition	CG Nicalon™	Manufacturer	COI
Ply Count	SiC	Structure/Architecture	8 harness satin weave fabric
Fiber Areal Weight	8	Ply Thickness	.346 mm
		Laminate Lay-up Sequence	0 <sub>r</sub> /90 <sub>r</sub> /0 <sub>r</sub> /90 <sub>r</sub> /90 <sub>r</sub> /0 <sub>r</sub> /90 <sub>r</sub> /0 <sub>r</sub>
<b>Matrix</b>	Si-C-N w/Si3N4 Particle Filler	Source	COI
<b>Interphase /Fiber Coating</b>			
Thickness	0.5µm	Composition	Boron Nitride
		Source	CVD
<b>External Coating</b>			
Composition	None	Thickness	na
Fabrication Method	na	Source	na
<b>Tensile Test Method</b>			
Test Method Spec.	ASTM C 1275	Test File ID#	na
Normalized by:		Date of Testing	May-Sept 1998
Modulus Calc Method	na	Load/Ext. Strain Rate	.02 mm/s
		P.L. Calculation Method	Extension under load

Date of Data Submittal

Mar-01

<b>Tension Test Data</b>	Temperature	23	
	Atmosphere	Air	
	Precondition	none	
	Gage Length	25 mm	
	Stress Orientation	0°	
		<b>Normalized</b>	<b>Measured</b>
Ultimate Tensile Strength $F_x^{tu}$ (MPa)	Mean		251
	S.Dev.		18.0
	# of Specimens		90
	# of Batches		6
	Approval Class		
Elastic Modulus $E_x^t$ (GPa)	Mean		93.0
	S.Dev.		4.65
	# of Specimens		90
	# of Batches		6
	Approval Class		
Prop. Limit Stress $F_x^{pl}$ (MPa)	Mean		85.0
	S.Dev.		3
	# of Specimens		90
	# of Batches		6
	Approval Class		
Strain at $F^{ut}$ $\epsilon_x^{tu}$ (µε)	Mean		4310
	S.Dev.		390
	# of Specimens		89
	# of Batches		6
	Approval Class		
Poisson's Ratio $\nu_{xy}^t$	Mean		
	S.Dev.		
	# of Specimens		
	# of Batches		
	Approval Class		

Approval Class = F - Fully Approved, I - Interim, S - Screening

Volume 5, Part D Data Requirements and Data Sets

<b>COMPOSITE</b>			
Composite Name	Sylramic S-200	Manufacturer	COI
Composite Description	SiC / BN / Si3N4	Manuf. Lot #	#14416
Reinforc. Vol %	45%	Date of Manufacture	Summer 1997
Density	2.2 g/cc	Fabrication Class	Preceramic polymer
% Porosity	2.7% Open	Dev/Prod. Status	Commercially Available
<b>Reinforcement</b>			
Composition	CG Nicalon™	Manufacturer	COI
Ply Count	8	Structure/Architecture	8 harness satin weave fabric
Fiber Areal Weight		Ply Thickness	.346 mm
		Laminate Lay-up Sequence	0 <sub>i</sub> /90 <sub>i</sub> /0 <sub>i</sub> /90 <sub>i</sub> /90 <sub>i</sub> /0 <sub>i</sub> /90 <sub>i</sub> /0 <sub>i</sub>
<b>Matrix</b>	<b>Composition</b>	Si-C-N w/Si3N4 Particle Filler	Source
			COI
<b>Interphase /Fiber Coating</b>		Composition	Boron Nitride
Thickness	0.5µm	Source	CVD
<b>External Coating</b>			
Composition	None	Thickness	na
Fabrication Method	na	Source	na
<b>Interlaminar Shear Test Method</b>		Test File ID#	na
Test Method Spec.	ASTM C 1292	Notch Distance	6 mm
Normalized by:		Date of Testing	Jul-Oct 98

Date of Data Submittal

Mar-01

<b>Shear Test Data</b>		Temperature	23°C
		Atmosphere	Air
		Precondition	none
		<b>Normalized</b>	<b>Measured</b>
Ultimate In-Plane Strength $F_{xy}^{su}$ (MPa)	Mean		33.0
	S.Dev.		5.00
	# of Specimens		70
	# of Batches		6
	Approval Class		

<b>Interlaminar Shear Test Data</b>		Temperature	23°C
		Atmosphere	Air
		Precondition	none
		<b>Normal-ized</b>	<b>Measured</b>
Ultimate In-Plane Strength $F_{yz}^{su}$ (MPa)	Mean		111
	S.Dev.		5.00
	# of Specimens		80
	# of Batches		6
	Approval Class		

Approval Class = F - Fully Approved, I - Interim, S - Screening

Volume 5, Part D Data Requirements and Data Sets

<b>COMPOSITE</b>			
Composite Name	Sylramic S-200	Manufacturer	COI
Composite Description	SiC / BN / Si3N4	Manuf. Lot #	#14416
Reinforc. Vol %	45%	Date of Manufacture	Summer 1997
Density	2.2 g/cc	Fabrication Class	Preceramic polymer
% Porosity	2.7% Open	Dev/Prod. Status	Commercially Available
<b>Reinforcement</b>	<b>CG Nicalon™</b>	Manufacturer	3M
Composition	SiC	Structure/Architecture	8 harness satin weave fabric
Ply Count	8	Ply Thickness	0.346 mm
Fiber Areal Weight		Laminate Lay-up Sequence	[0/90/0/90/90/0/90/0]
<b>Matrix</b>	Si-C-N w/Si3N4 Particle Filler	Source	COI
<b>Interphase /Fiber Coating</b>		Composition	Boron Nitride
Thickness	0.5µm		CVD
<b>External Coating</b>			
Composition	none	Thickness	na
Fabrication Method	na	Source	na
<b>Flexure Test Method</b>		Test File ID#	
Test Method Spec.	ASTM C 1341	Geometry (3 Pt/4 Pt)	4 pt
Outer Span Length( mm)	80 mm	Span/Depth Ratio	29.2
Modulus Calc Method	na	P.L. Calculation Method	na
Normalized by		Date of Testing	Dec 97 - Apr 1998

Date of Data Submittal **Mar-01**

<b>Flexure Test Data</b>	Temperature	23	
	Atmosphere	Air	
	Precondition	none	
	Stress Orientation	0°	
		<b>Normalized</b>	<b>Measured</b>
Ultimate Flexural Strength $F^{bu}$ (MPa)	Mean		339
	S.Dev.		37.0
	# of Specimens		100
	# of Batches		6
	Approval Class		
Elastic Modulus $E^b$ (GPa)	Mean		93.0
	S.Dev.		6.00
	# of Specimens		100
	# of Batches		6
	Approval Class		
Prop. Limit Stress $F^{pl}$ (MPa)	Mean		
	S.Dev.		
	# of Specimens		
	# of Batches		
	Approval Class		
Strain at $F^{bu}$ $\epsilon^{bu}$ (µε)	Mean		4640
	S.Dev.		630
	# of Specimens		100
	# of Batches		6
	Approval Class		
Poisson's Ratio $\nu_{xy}^b$	Mean		
	S.Dev.		
	# of Specimens		
	# of Batches		
	Approval Class		

Approval Class = F - Fully Approved, I - Interim, S - Screening

**DEPARTMENT OF DEFENSE  
HANDBOOK**

**CERAMIC MATRIX COMPOSITES**

**VOLUME 5**

**APPENDIX A – DERIVATION OF THE RESIDUAL  
STRENGTH REDUCTION EXPRESSIONS FOR LCF  
AND RUPTURE LOADINGS**

**APPENDIX A. DERIVATION OF THE RESIDUAL STRENGTH REDUCTION  
EXPRESSIONS FOR LCF AND RUPTURE LOADINGS**  
(Equations: 6 through 11)

The major premise of the approach is that the residual strength,  $X$ , of a material degrades as a function of stress and cycles for fatigue and a function of stress and time for rupture. Damage initiation is predicted when the residual strength or remaining strength equals the applied stress. For this write-up, residual strength and remaining strength are used interchangeably. Figure A.1 contains a residual strength curve as a function normalized cycles for a theoretical CMC under constant fatigue loading. Based on experience, the residual strength reduction in Figure A.1 is typically nonlinear and can be reasonably modeled by an expression of the form

$$X = X^0 - [X^0 - \sigma_r] * \left(\frac{n}{N}\right)^p \quad (\text{A.1})$$

where,  $X$  is the residual strength of the material,  $X^0$  is the initial strength,  $\sigma_r$  is the stress range,  $n$  is the number of cycles at  $\sigma_r$ ,  $N$  is the number of cycles to failure at  $\sigma_r$  from an S-N curve, and  $p$  is the residual strength shape parameter for fatigue. For  $p > 1$ , the shape of the residual strength curve is concave down as shown. We can normalize (A.1) with respect to the initial strength of the material,  $X^0$ , to get

$$R = 1 - \left[1 - \frac{\sigma}{X^0}\right] * \left(\frac{n}{N}\right)^p \quad (\text{A.2})$$

where  $R$  is the normalized residual strength. Differentiating (A.2) with respect to  $n$ , we get

$$dR = 1 - \left[1 - \frac{\sigma}{X^0}\right] * p \left(\frac{n}{N}\right)^{p-1} d\left(\frac{n}{N}\right) \quad (\text{A.3})$$

If we assume  $\sigma$  is not constant over the life of a part, we need to sum up all of the differential  $dR$ 's for different  $\sigma_r$  in order to get  $R$ . In mathematical terms, we have

$$R = 1 - \int \left[1 - \frac{\sigma}{X^0}\right] * p \left(\frac{n}{N}\right)^{p-1} d\left(\frac{n}{N}\right) \quad (\text{A.4})$$

For complex loadings, we can replaced  $\sigma$  with  $\sigma_r$  which has been corrected for mean stress and TMF effects. At this point, let us return to equation (A.2) and substitute  $\sigma_r$  for  $\sigma$  to get

$$R = 1 - \left[1 - \frac{\sigma_r}{X^0}\right] * \left(\frac{n}{N}\right)^p \quad (\text{A.5})$$

Now consider a load history which consists of  $\Delta n_1$  cycles at  $\sigma_1$  and  $\Delta n_2$  cycles at  $\sigma_2$ . Assume  $\sigma_1 > \sigma_2$ . We would like to determine the residual strength,  $R_2$ , after  $\Delta n_1 + \Delta n_2$  cycles. The residual strength calculations are show graphically in Figure A.2. For the initial  $\Delta n_1$  cycles, the strength degrades along line A-B. Using equation (A.5), the residual strength after  $\Delta n_1$  cycles is give as

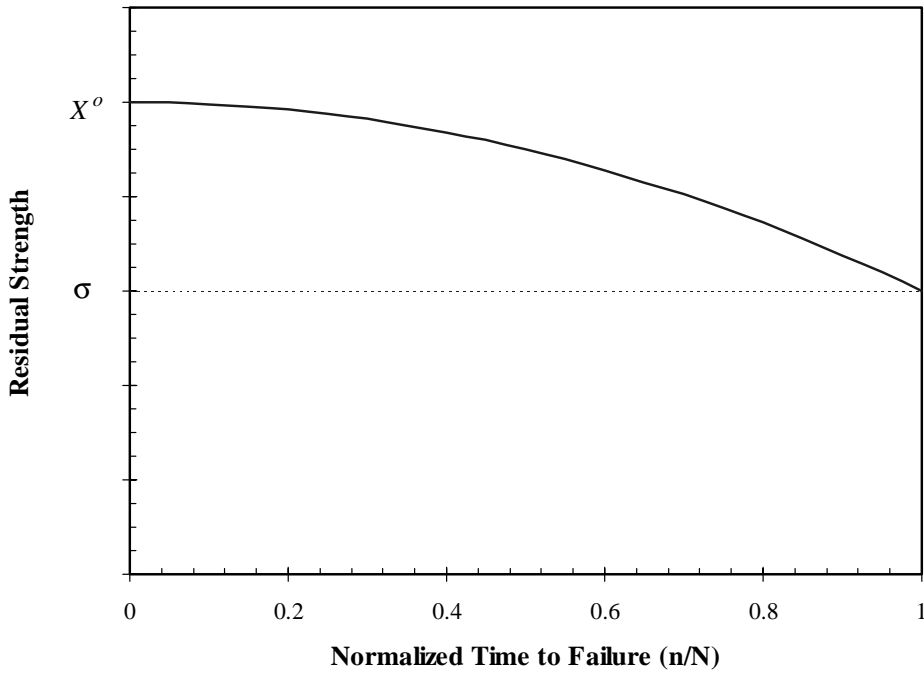


FIGURE A.1. Residual strength curve as a function of normalized cycles for  $P=2.0$ .

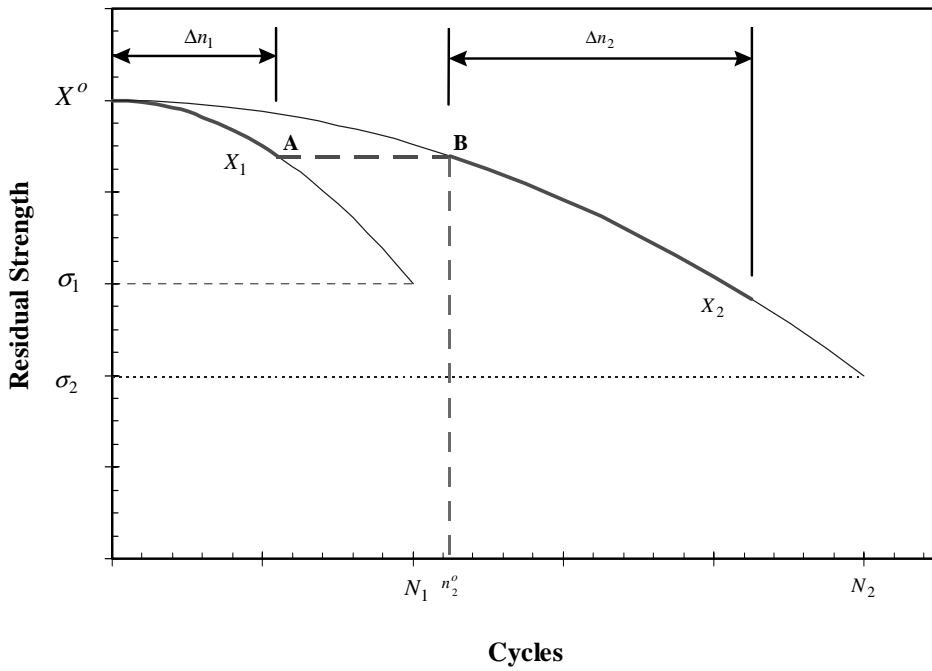


FIGURE A.2. Residual strength reductions for a two step load history.

$$R_1 = 1 - \left[ 1 - \frac{\sigma_1}{X^0} \right] * \left( \frac{\Delta n_1}{N_1} \right)^p \quad (\text{A.6})$$

and

$$X_1 = R_1 * X^0 \quad (\text{A.7})$$

In order to shift from the  $\sigma_1$  stress to the  $\sigma_2$  stress, we need to move along the dotted line B-C, and we must calculate the equivalent cycles  $n_2^0$  at Point C for the current residual strength. Based on a concept of equivalent residual strength and equation (A.5), we can also write

$$R_1 = 1 - \left[ 1 - \frac{\sigma_2}{X^0} \right] * \left( \frac{n_2^0}{N_2} \right)^p \quad (\text{A.8})$$

Rearranging this equation, we have

$$n_2^0 = N_2 \left[ \frac{1 - R_1}{1 - \frac{\sigma_2}{X^0}} \right]^{\frac{1}{p}} \quad (\text{A.9})$$

where  $R_1$  is determined from equation (A.6). Next, we need to continue the strength degradations along the line C-D for  $\Delta n_2$  cycles, and the residual strength at point D,  $R_2$ , is given by

$$R_2 = 1 - \left[ 1 - \frac{\sigma_2}{X^0} \right] * \left( \frac{n_2^0 + \Delta n_2}{N_2} \right)^p \quad (\text{A.10})$$

subtracting equation (A.8) from (A.10), we get

$$\Delta R_2 = - \left[ 1 - \frac{\sigma_2}{X^0} \right] * \left\{ \left( \frac{n_2^0 + \Delta n_2}{N_2} \right)^p - \left( \frac{n_2^0}{N_2} \right)^p \right\} \quad (\text{A.11})$$

$$R_2 = R_1 - \Delta R_2 \quad (\text{A.12})$$

and

$$X_2 = R_2 * X^0 \quad (\text{A.13})$$

In generalized form, we can write equations (A.9), (A.11), and (A.12) for the  $i$ 'th cycle as

$$n_i^0 = N_i \left[ \frac{1 - R_{i-1}}{1 - \frac{\sigma_i}{X^0}} \right]^{\frac{1}{p}} \quad (\text{A.14})$$

$$\Delta R_i = - \left[ 1 - \frac{\sigma_i}{X^0} \right] * \left\{ \left( \frac{n_i^0 + \Delta n_i}{N_i} \right)^p - \left( \frac{n_i^0}{N_i} \right)^p \right\} \quad (\text{A.15})$$

$$R_i = R_{i-1} - \Delta R_i \quad (\text{A.16})$$

and

$$X_i = R_i * X^0 \quad (\text{A.17})$$

We can perform the same development for rupture loadings which will yield essentially the same equations shown above except  $n$ ,  $N$ ,  $p$ ,  $k$  get replaced by  $t$ ,  $T_{rup}$ ,  $q$ , and  $j$ , respectively, where  $t$  is time,  $T_{rup}$  is the rupture life,  $q$  is the rupture shape parameter, and  $j$  is load step number. These rupture equations are as follows

$$t_j^0 = T_{rup,j} \left[ \frac{1 - R_{j-1}}{1 - \frac{\sigma_j}{X^0}} \right]^{\frac{1}{q}} \quad (\text{A.18})$$

$$\Delta R_j = - \left[ 1 - \frac{\sigma_j}{X^0} \right] \left\{ \left( \frac{t_j^0 + \Delta t_j}{T_{rup,j}} \right)^q - \left( \frac{t_j^0}{T_{rup,j}} \right)^q \right\} \quad (\text{A.19})$$

$$R_j = R_{j-1} - \Delta R_j \quad (\text{A.20})$$

and the degraded strength,  $X_j$ , can be calculated as

$$X_j = R_j * X^0 \quad (\text{A.21})$$

Damage initiation is predicted when the applied stress exceeds the residual strength or

$$\sigma \geq X \quad (\text{A.22})$$

Equations (A.14) through (A.22) represent the heart of the residual strength approach. These equations are relatively straightforward and can be implemented into computer programs to address complex loading histories.



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