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Design Methodology Needs for Fiber-Reinforced Ceramic Heat Exchangers

J. J. Blass
M. B. Ruggles

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Engineering Technology Division

DESIGN METHODOLOGY NEEDS FOR FIBER-REINFORCED CERAMIC HEAT EXCHANGERS

J. J. Blass
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SUMMARY

An initiative undertaken by the U.S. Department of Energy, Office of Industrial Programs, is to foster the development and commercialization of high-pressure heat-exchange systems (HiPHES). The HiPHES program is based upon rapidly developing new materials, including fiber-reinforced ceramic composites, that offer greater strength at high temperature and in corrosive environments than current materials. Previously a panel of process/industry experts assessed the potential of HiPHES and identified major opportunities for development of advanced, high-efficiency processes in power generation, steam reforming of natural gas, evaporation of corrosive chemicals, and heat recovery from corrosive waste gases. Consequently a development program was recommended, and a study was suggested to identify significant development and validation needs that must be satisfied to establish a viable design methodology for ceramic-fiber, ceramic-matrix composite materials in advanced heat exchangers.

A panel of experts with government, university, and industry affiliations conducted the present study. As in a previous materials assessment performed for the HiPHES program, general agreement was that fiber-reinforced ceramics technology is immature and that a sufficient data base does not exist to properly assess the performance and reliability of these materials, especially for long-term service. To identify design needs for fiber-reinforced ceramics, the panel recognized two types of design involved, design <u>of</u> the material and design <u>with</u> the material, and tried to focus primarily on the latter. Despite this focus, considerable attention was paid to micromechanical modeling of fiber-reinforced composites as a means of assessing the factors that determine macroscopic behavior.

The present report reviews important ingredients of structural design – analysis methods and design criteria. Mechanical modeling techniques specific to composite materials and structural modeling approaches are discussed. The concept of probabilistic failure analysis is also introduced. Design criteria are further discussed with emphasis on the failure mechanisms of ceramic composites. Concepts of reliability and risk assessment resulting from design methodologies are addressed. Finally, research activities of other federally funded agencies in the area of fiber-reinforced ceramic-matrix composites are surveyed. As a result, the following design methodology development and validation needs for fiber-reinforced ceramic heat exchangers are identified:

1. realistic representation of the effective anisotropic stiffness properties of these materials as influenced by temperature, time, loading cycles, environment, and state of stress;
2. extensive testing, examination, and analytical modeling to develop an understanding of the factors that influence stiffness and strength, in particular, the variable characteristics of the interface between fiber and matrix;
3. adaptation of finite-element analysis techniques to account for different stiffness values in tension and compression;
4. analytical models of inelastic behavior under off-normal conditions;
5. special testing techniques suitable for the unique characteristics of these materials;
6. comprehensive failure criteria based on realistic models of material behavior under short-term, long-term, and cyclic loading conditions; and
7. structural tests and analyses to confirm the validity of the overall methodology.

1. INTRODUCTION

The purpose of this report is to identify significant development and validation needs that must be satisfied to establish a viable design methodology for use of ceramic-fiber-reinforced, ceramic-matrix composite materials in advanced heat exchangers for high-temperature and high-pressure applications. This is part of an initiative undertaken by the U.S. Department of Energy (DOE), Office of Industrial Programs (OIP), to foster the development and commercialization of high-pressure heat-exchange systems (HiPHES). As described in the next section, previous studies identified several industrial applications of HiPHES technology in which the use of fiber-reinforced ceramic composites can provide very significant benefits. The studies resulted in a recommended development program that includes an early assessment of design methodology needs for these materials and applications. To provide this assessment, Oak Ridge National Laboratory (ORNL) assembled a panel of experts with government, university, and industrial affiliations. The present panel met in April 1988 for presentations and discussions related to structural design methodology for fiber-reinforced ceramic heat-exchanger components. This report is based, in part, on those discussions and on written contributions subsequently provided by panel members.

In terms of report content and organization, eight major sections follow the background sections. Chapter 3 defines and reviews the key ingredients of structural design that are categorized broadly as analysis methods and design criteria. Next, in Chap. 4, the mechanical modeling techniques/approaches that are necessary and distinctive for composite materials and structural modeling are reviewed. Chapter 5 elaborates on analysis methods at the material constitutive relationship and spatial discretization levels and discusses the possible complications due to off-normal operating conditions. The concept of probabilistic failure analysis is also introduced. In Chap. 6, design criteria are further discussed with much attention to the failure mechanisms of ceramic composites. Concepts of reliability and risk assessment resulting from design methodologies are addressed. Then, Chap. 7 introduces

the subject of validation of methods and criteria through structural testing and comparison analysis. Chapter 8 lists the development and validation needs in summary and in categories to add specificity. Chapter 9 provides the recommended approach to meeting the needs delineated in Chap. 8. Finally, in Chap. 10, research activities in the area of fiber-reinforced ceramic-matrix composites are surveyed in other federally funded agencies.

2. BACKGROUND

The DOE OIP is engaged in an effort to foster the development of advanced HiPHES, for subsequent commercialization by U.S. industry. Use of these systems could result in estimated annual savings of 0.5 quad in energy consumption and $2.1 billion in costs,[1] while providing higher process yields and greater latitude in fuel selection. The HiPHES program is based upon rapidly developing new materials technologies, including fiber-reinforced ceramic composites. These new materials offer greater strength at high temperature and in corrosive environments than current materials, without reliance on strategic materials.

The potential of HiPHES was assessed first by a panel of process/industry experts[1] who identified major opportunities for development of advanced, high-efficiency processes in power generation, steam reforming of natural gas, evaporation of corrosive chemicals, and heat recovery from corrosive waste gases. Based upon these potential applications and a preliminary definition of design requirements for each[1] (Table 1), a second panel of materials experts made the following recommendations:[1]

1. Institute design-oriented studies to define and document detailed materials properties, as well as detailed application requirements and manufacturing methods to construct bench-scale or larger prototype models of the promising applications.
2. Conduct applied research in the areas of physical properties and corrosion/environmental effects involving identified industrial process fluids and conditions on candidate materials for heat-exchange systems.
3. Develop quality assurance, acceptance criteria, and standards principally using nondestructive detection and evaluation techniques to meet engineering design codes and methodologies.
4. Because of the significant amount of R&D in high-strength, high-temperature materials for nonindustrial applications (military and space technology, notably), maintain an active OIP technology liaison with other federal agencies and their contractors.

Table 1. Summary of HiPHES application design requirements

Attribute	Application			
	Steam reformers	Power generation	Chemical evaporators	BOF[a] hood
Geometry and dimensions	Tubular, 2-in. ID, and 32 ft long	Tubular, 1- to 2-in. ID, and up to 40 ft long	Tubular, 1.8-in. ID, and 16 to 24 ft long	Tubular/membrane panels, 1- to 2-in. ID, and 10 to 40 ft or longer
Maximum working temperature, °F	1900	2700-3000	350	500
Maximum working pressure differential, psi	950	300-900	170	720
Fluid constituents and materials compatibilities	Methane, water vapor, hydrogen, carbon monoxide, carbon dioxide	Water and water vapor, sulfur compounds, ash particulates, calcium and magnesium compounds	50% concentrated acidic and basic solutions, scaling tendencies with metals	Halogens; acids; sodium and calcium silicate, sulfate and carbonate salts at 1000-1500°F; metal slags to 3,000°F
Mechanical stresses and vibrations	Internal static loads, no appreciable vibration	Prmarily internal static loads, no extraordinary vibrations under normal conditions	Primarily static loads, no appreciable vibration	Substantial vibration, tubes must hold steam and boiling water to 720 psia
Thermal expansion, in./in.-°F	10×10^{-6}	9 to 10×10^{-6}	5.5 to 7.5×10^{-6}	6×10^{-6}
Thermal shock	150°F in 1 h	600°F in 5 min	Small temperature differences, process limited to relatively slow changes	1500 to 3500°F in 10 min

Table 1 (continued)

Attribute	Application			
	Steam reformers	Power generation	Chemical evaporators	BOF[a] hood
Porosity and safety considerations	Must be virtually zero, must meet pressure vessel codes	Leakage caused by poor seals and material porosity parasitic to cycle efficiency and deleterious to plant objectives, must meet pressure vessel codes	No leakage or porosity, must meet pressure vessel codes	Leakage and porosity close to 0, must meet pressure vessel codes
Service life and serviceability	5 to 12 years, retrofit capability	Semiannual inspection and cleaning, expect one or two tubes to be replaced annually	3 to 8 years for tubes, must be roddable, retrofit capability	5 years or more, replaceable, retrofit capability
Thermal conductivity, Btu/ft^2-h-°F	45	10 to 20	42	26

[a]Basic oxygen furnace.

These recommendations were derived from requirements for potential industrial applications and gaps in capabilities of current and emerging materials. The most promising materials for the long term are ceramic composites because of their ability to maintain strength at high temperatures. The most promising industrial applications are in power generation, using high-temperature dirty or waste gases, and in steam reforming of methane-based gases used to produce hydrogen. Nevertheless, it was possible to identify generic R&D areas common to all the applications based on preliminary design requirements identified in the study.

Figure 1 shows the recommended time-based program[1] that integrates the four R&D requirements. Design needs and methodologies, along with further identification of detailed application requirements, merge with studies of candidate materials and their manufacturing and quality control methods to provide the basis for first-generation HiPHES prototype tests. The development of acceptance criteria and knowledge of additional materials would precede second-generation testing.

The materials experts were able to identify several problem areas pertinent to HiPHES applications.[1] Some of the more critical follow:

1. Ceramic composite materials technology is in its infancy and has an insufficient data base for assessment and evaluation purposes, such as the ability to meet the HiPHES specifications.
2. Long-term testing of advanced materials in actual environments has not been adequately conducted. Issues such as creep, tensile strength, embrittlement, and catastrophic failures need to be evaluated.
3. The interface and synergy between the matrix and fiber/whisker components of composite materials is an important consideration that has not been completely resolved.
4. Monolithic ceramics and composite materials currently have significantly higher total costs than conventional materials. This will need to be addressed during any R&D program and is not part of the HiPHES study.

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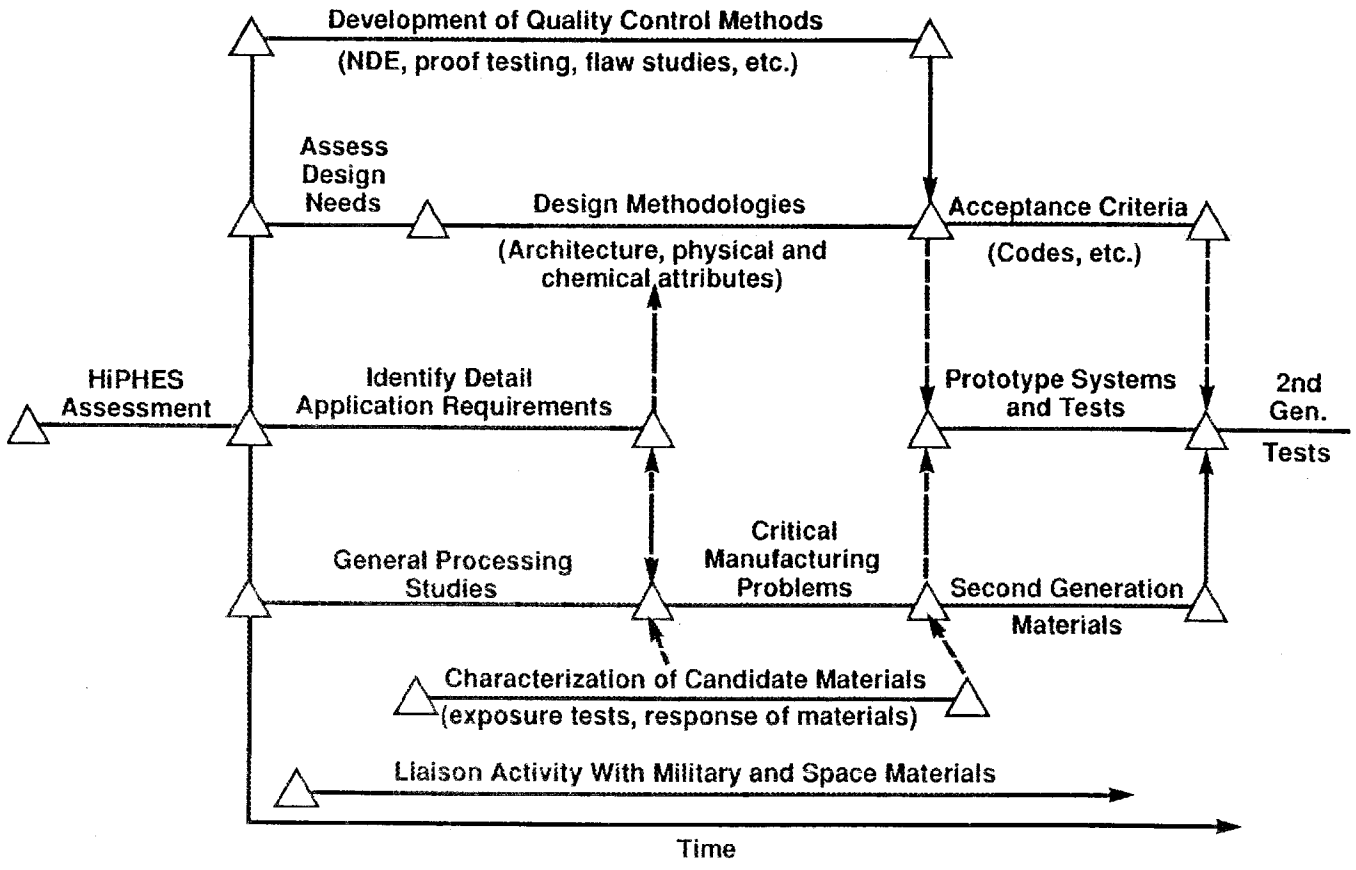

Fig. 1. Recommended HiPHES development program. *Source:* S. A. Richlen, "Assessment of High Pressure Heat Exchange Systems Technology," presented at the Department of Energy Advanced Heat Exchangers Program Review, October 28-29, 1987.

5. Metals and refractory metals should not be precluded from HiPHES consideration; some of them appear attractive in terms of high-temperature applicability for the proposed HiPHES applications. However, other considerations such as cost, availability (strategic), and manufacturability can impede their use; such issues have not been resolved. In addition, considerable R&D activity for these materials is taking place in the private sector.

3. KEY INGREDIENTS OF STRUCTURAL DESIGN METHODOLOGY

In its deliberations, the present panel was sensitive to the great many design criteria that are not strictly related to mechanical (stress or strain) limits. For instance, the corrosion and erosion resistance of the composite material in these applications is a significant design consideration, as is the chemical compatibility of the constituents of the composite material with each other and with other elements of the system at elevated temperature. The thermophysical properties (thermal conductivity, heat capacity, and thermal coefficient of expansion) have a significant influence on the thermal efficiency of the heat exchanger as well as on the thermally induced stresses and strains in the material. Because the panel was convened to deal with mechanical aspects of the design methodology, these other design considerations have not been analyzed in detail. However, they may be the limiting factors in system performance and thus are extremely important in the materials selection and materials design stage of the design process.

To identify design needs for ceramic composites, the panel recognized two types of design involved – design <u>of</u> the material and design <u>with</u> the material. Design of the material refers specifically to (1) selecting the constituents using thermodynamic and micromechanical models; (2) tailoring the interface using additives, coatings, etc., to achieve the desired strength and stability of bonding; (3) choosing processing techniques for the composites; (4) specifying the fiber architecture [e.g., laminates, weaves, braids, knits, and three-dimensional (3-D) assemblies]; and (5) selecting a fabrication technique for the component. This process must be done with full appreciation of the expected service conditions for the component. In terms of the traditional design process for monolithic ceramics or metals, this step replaces the materials selection step, which usually precedes the structural design step. The panel concentrated its attention on structural design methodology or designing with the material. However, in designing with composite materials certain elements <u>of</u> the design of the material, (e.g., fiber orientation and volume fraction) must be included to create the most efficient design for the component. Therefore, the

detailed discussion of the design methodology presented below does include variables associated with design of the material.

In the design of a structural component, the principal objectives are to ensure that functional, economic, and structural integrity requirements are satisfied throughout the useful life of the component. Achieving these objectives generally requires mature materials and structural design technologies. Regardless of the structural material employed, the principal ingredients of structural design methodology fall into two broad categories: analysis methods and design criteria. These are discussed in the next two sections.

3.1 ANALYSIS METHODS

Analysis methods are the procedures used to calculate components of stress and strain throughout a body from the loads and displacements (both mechanical and thermal) imposed on it during the time period of interest. The principles of equilibrium between stresses and loads and of compatibility between strains and displacements must be satisfied; but an especially significant ingredient of the analysis methods is the constitutive model or the relationship between stresses, strains, temperature, and time that describes the behavior of the material.

Real material behavior is complicated, so idealizations are employed in structural design analyses. For most of their useful range of application, fiber-reinforced ceramics are expected to be idealized as linear elastic, nominally requiring that only stiffness and thermal expansion properties be specified as a function of temperature. (This is easier said than done, for a host of reasons, not the least of which is the heterogeneous nature of composite materials, as discussed below.) Within the framework of the elastic idealization, certain features of real behavior, such as internal damage accumulated during operation, can be at least partially accounted for by using reduced stiffness values.

3.2 DESIGN CRITERIA

Design criteria constitute the other broad category of structural design ingredients. In the present context, the most significant design criteria are those that, in effect, establish limiting values, which may not be exceeded by the stresses or strains calculated using the analysis methods discussed above. Typically the limits are set low enough to provide suitable margins against anticipated modes of failure, where failure is broadly defined as loss of, or significant reduction in, the capability to perform a required function. For a high-pressure heat exchanger, a certain amount of fluid leakage through relatively small openings might constitute failure. If the material on the pressure boundary is a ceramic-fiber, ceramic-matrix composite, significant leakage paths could be formed by linkage of many small cracks and defects in the matrix and the fiber-matrix interface at stress levels much less than the strength of the fiber bundle.

The stress limits used in design obviously depend on the strengths of the materials employed. However, the margins against failure depend largely on the uncertainties in stress and strength, as well as on the expected consequences of failure. The uncertainty in stress depends on the accuracy of the load predictions and on the accuracy of the analysis methods, which for ceramic composites depends on the accuracy of the stiffness properties employed. The uncertainty in strength for most materials depends on variations in material and processing; but, for ceramic composites, other issues are equally important as discussed in Chap. 6.

Many of the stiffness and strength properties of fibrous composites cannot be measured directly but must be estimated based on the results of analyses and simple tests as discussed in the next chapter.

4. MECHANICAL MODELING OF COMPOSITE MATERIALS AND STRUCTURES

Most common engineering materials are homogeneous and isotropic, meaning that their mechanical (e.g., stiffness and strength) properties are independent of position and direction. Fiber-reinforced composite materials are both heterogeneous and anisotropic, requiring that they be studied from two points of view: the microscale and the macroscale. Micromechanics is the study of the mechanical behavior of composite materials in which the interaction of the constituent materials is examined. Macromechanics is the study of composite behavior in which the material is presumed to be homogeneous, and the influence of the constituent materials is felt only as averaged overall properties of the composite. Because of the directional nature of fiber reinforcement, this quasi-homogeneous material remains anisotropic, requiring a greater number of parameters to describe its macroscopic behavior than for an isotropic material. Micromechanics aims at understanding of the fundamental mechanisms that determine composite behavior and can provide a basis for estimating macroscopic properties. Macromechanics can serve as a practical basis for design analysis of structural components.

The finite-element method is now widely used for both micromechanical and macromechanical analyses. Specialized computer codes[2,3] have been developed to permit detailed analysis of a representative unit cell of composite material with complicated 3-D fiber architecture. Such codes accept anisotropic, temperature-dependent properties of the constituent materials; they are compatible with interface modeling techniques that permit changing connectivity and force conditions between elements; and they are capable of performing automated failure analyses. The results of micromechanical analyses can be used to estimate macromechanical properties of composites with complex fiber architecture based on results of tests of unidirectionally reinforced or other simple composites. The macromechanical properties are used in general-purpose, finite-element computer codes to perform design analyses of structural components with complicated geometries. A number of commercially available codes, such as SAAS, ABAQUS, MARC, ANSYS, PATRAN, and NASTRAN, will accept orthotropic or fully anisotropic thermoelastic material

properties. Such codes contain extensive libraries of finite elements including 2-D and 3-D solid elements, and special plate and shell elements. The solid elements can be used with anisotropic material properties, and the plate and shell elements can be used to represent laminated composites.

5. ANALYSIS METHODS

One of the most significant ingredients of analysis methods is the constitutive relationship between stresses, strains, temperature, and time that describes the mechanical behavior of the material. For most of their useful range of application, fiber-reinforced ceramics can be idealized as linear elastic. Although these materials can be regarded as quasi-homogeneous on the macroscale, the directional nature of their fiber reinforcement must be taken into account. As anisotropic materials, more stiffness and thermal expansion properties are required to describe their behavior than are required for isotropic materials. Although many of these properties can be measured directly through testing of unidirectionally reinforced or other simple specimens, micromechanical analysis provides invaluable insights into parameters governing behavior of composites.

5.1 MICROMECHANICAL MODELING

5.1.1 Micromechanics—Description of the Overall Composite Properties Through the Properties of Microscopic Constituents

Micromechanics represents an approach within which the overall properties of composite materials are related to the behaviors and interactions of their microscopic constituents, such as grains, particles, and fibers. For example, elastic moduli of the composite can be represented as functions of the elastic moduli of matrix and fibers and the volume fraction of fibers. Concentric cylinder models, which recognize a fairly realistic cylindrical fiber geometry, are useful tools for estimating the overall properties of fibrous composites.

For some composite materials, microlevel geometry must be incorporated into the micromechanical models. Various textile preforms, such as braided or woven fabrics, are under consideration as candidate fiber architectures for fiber-reinforced ceramic composites. To accurately model the structural features of such a composite as a quasi-homogeneous, anisotropic solid, it is necessary to develop a Fabric Geometry Model (FGM).[4] Establishment of an FGM consists of

(1) identification of the unit cell geometry, (2) specification of the fiber architecture (or fabric geometry) within the unit cell, and (3) incorporation of the constituent material properties by means of a micromechanical model of the unit cell.

Because properties of microscopic constituents depend on various factors such as temperature, time, cycles, and environment, the overall properties of a fiber-reinforced ceramic composite will depend upon the same factors. In addition, the properties of a composite are influenced by the nature of the interface between fiber and matrix. Tremendous differences between the limiting cases of perfect continuity and complete lack of continuity at the interface were shown to occur both theoretically and experimentally. The sign (and possibly the magnitude) of the transverse normal stress components and the nature of the residual (curing) stress field are the governing parameters responsible for the dependence of the composite properties on the fiber-matrix interface. At present, fundamental understanding of the influence of the interface on material performance is lacking. Therefore realistic representation of the anisotropic properties of the composite may require extensive material characterization testing under a variety of environmental and loading conditions. Combined analytical and experimental approaches guided by considerable engineering judgment are expected to be instrumental in establishing a realistic material representation.

5.1.2 Micromechanical Failure Modeling

Micromechanical failure modeling represents another direction in micromechanics. In this case the overall failure of the composite is related to failure on the microlevel, that is, fiber failure, matrix cracking, etc. Several analytical techniques were employed to characterize microlevel failure. Among these are the 2-D and 3-D shear-lag models introduced by Cox.[5] Within the shear-lag model framework, the axial load capacity of the matrix material is neglected; the fibers are assumed to bear all the axial load in tension, while the matrix carries only shear stress. While at present none of these models can accurately describe the response of the ceramic matrix composites, they can be

generalized to represent such materials.[6] Another failure model of particular interest was employed by Marshall et al.[7,8] In Ref. 7 the authors examined composites that exhibit multiple matrix cracking before fiber failure in the presence of purely frictional bonding between the fibers and matrix. A fracture mechanics approach with cracking criteria based upon stress-intensity considerations was employed to evaluate the stress for matrix cracking. The influence of the fibers bridging the matrix crack was represented by closure tractions at the crack surfaces. Long and short cracks were distinguished. In Ref. 8 the stress-intensity analysis was generalized to include the case of fiber failure occurring behind the crack tip. This analysis allowed (1) crack-growth behavior to be evaluated for two distinct mechanisms of failure and (2) the failure conditions and transitions between the two mechanisms to be related to microstructural properties of the composite.

Another example of a microfailure model is given in the work of Aveston et al.[9,10] The authors considered the onset of matrix cracking in the presence of (1) slip along the entire fiber-matrix interface (large slip), (2) a continuous fiber-matrix bond (no slip), and (3) partial slip (combination of large slip and no slip). Investigation was limited to the case of debonding caused by shear stress only.

Budiansky et al.[11] formulated a microfailure model, within which a general energy relationship was developed for a class of steady-state fracture problems, including the effects of friction and initial stress. It is recognized that a fiber-reinforced ceramic composite subjected to tensile loading in the direction of the fibers can undergo extensive matrix cracking normal to the fibers, the fibers remaining intact. Budiansky et al.[11] studied the critical conditions for the onset of widespread matrix cracking on the basis of fracture mechanics theory. Two situations at fiber-matrix interface were considered: (1) unbonded fibers initially held in the matrix by strain mismatch, but susceptible to frictional slip, and (2) initially weakly bonded fibers, which may be debonded by the stresses at the tip of the matrix crack. Optimal thermal strain mismatches for maximum cracking strength (the magnitude of the initial fiber-matrix strain mismatch that leads to the maximum value of the applied stress at steady-state matrix cracking) were studied. A

formulation for the debonding of fibers under radial stress was provided as well. The results and conclusions of Budiansky et al.[11] derived from fracture mechanics concepts resemble those reached earlier by Aveston et al.,[9,10] which were based on somewhat more elementary considerations.

5.2 MACROMECHANICAL MODELING

As previously discussed, design analyses of structural components are typically performed using finite-element techniques to calculate values of stresses and strains throughout the component caused by the expected thermal and mechanical loadings. The results of these analyses are used to show that design limits are satisfied and that adequate margins are present to guard against known modes of failure. In these analyses, fiber-reinforced composite materials are usually treated as quasi-homogeneous, anisotropic, linear elastic solids. (For laminated composites, this applies to each layer of material; thus, special plate and shell elements are usually employed.) Most commercially available finite-element computer codes accept orthotropic and fully anisotropic thermoelastic properties, that is, arrays of temperature-dependent stiffness and thermal expansion coefficients. For a given composite material, these properties are obtained from micromechanical analyses and tests of simple composite specimens.

For some brittle-matrix composites, the results of tests and micromechanical analyses indicate that, because of fiber-matrix interaction, the macroscopic stiffness values depend strongly on the normal stresses acting perpendicular to the direction of the fibers. Different stiffnesses may be required for tensile values of these stresses than for compressive values. For realistic component geometries and histories of loading, this complicates what would otherwise be a fairly routine solution of a linear finite-element problem, because the sign of these stresses is unknown until the problem is solved. Iterative methods of solution must be employed for this sort of nonlinear problem much the same as for other problems involving material or geometrical nonlinearities.[12]

5.2.1 Laminate Model

Laminate modeling for certain classes of composite materials, such as fiber-reinforced graphite-epoxy, is a well-developed and demonstrated technology. In a laminate model, each layer of composite material is regarded as a quasi-homogeneous, anisotropic lamina. Except to represent a delaminated structure, the layers are assumed to be continuous at all interfaces. Detailed finite-element representations and also various levels of approximation are employed according to the accuracy desired. Good agreement exists between theoretically predicted and experimentally measured values of laminate stiffness. Usually, but not always, special problems such as plate vibrations and buckling are treated with sufficient accuracy by means of simplified models based on the effective properties of the composite as a whole.

It is expected that the existing laminate models can be employed, at least as a first approximation, in the description of fiber-reinforced ceramic laminates. The issues that must be settled before final acceptance of this approach are related to (1) representation of a poorly bonded fiber-matrix layer as a homogeneous, anisotropic lamina and (2) establishment of a self-consistent formulation for estimating effective stiffness and a methodology for predicting failure mode. The use of different values of elastic moduli under tension and compression is expected to be required, where the relevant components of stress are the normal stresses acting perpendicular to the fibers in each layer. This issue derives from the expected variability in the characteristics of the fiber-matrix bond as functions of applied stress state, residual stress state, and environmental conditions.

5.3 DEVIATIONS FROM NORMAL OPERATING CONDITIONS

While normal operating conditions will be of primary concern in the design of heat-exchanger components, consideration must also be given to deviations from normal conditions. For heat exchangers, examples of such deviations include increases in operating temperature, pressure, and concentration of corrosive products in the gas stream.

Such conditions either increase the stress on structural materials or reduce their stiffness and strength. Under these conditions, the response of the materials may depart substantially from a linear elastic behavior and more complex models of material behavior may be required for structural analyses.

5.4 PROBABILISTIC METHODS

Analysis methods for ceramics and composites have been hampered by inherent scatter in experimental failure data. The Weibull weakest link failure theory[13] has been applied to the design of monolithic ceramics for scaling laboratory specimen test results to determine ceramic component strengths.[14] More recently, these methods have been further developed through the incorporation of fracture mechanics concepts.[15,16]

Traditional (organic matrix) composites have also exhibited scatter in strength properties.[17] Likewise, some of the fibers being developed for ceramic-matrix composites exhibit (single-filament) strengths in inverse proportion to the test gage length,[18] suggesting weakest link type behavior. Extension of the deterministic methods described in the preceding sections are being investigated as a means of including statistical scatter in the design methodology.

6. DESIGN CRITERIA

The design criteria of interest are those that establish stress or strain limits low enough to provide adequate reliability against anticipated mechanisms of failure. Fracture or other structural failures are not the only mechanisms of interest for heat-exchange equipment components. Formation of leakage paths by linkage of cracks and defects that lead to excessive permeability can also constitute failure.

Design criteria for monolithic structural ceramics have traditionally been based on statistical reliability concepts because of their wide variation in failure strengths. Likewise, traditional (organic matrix) composites exhibit considerable variation in failure strength. It is therefore reasonable to assume that the scatter in performance properties exhibited by ceramic composites compounds the variability that is inherent in the bulk ceramic materials with the statistics of fiber strengths and fiber/matrix interfacial characteristics.

6.1 FAILURE MECHANISMS

An understanding of the failure mechanisms of ceramic-fiber, ceramic-matrix composites for structural applications at high temperature is primitive at best. Understanding of the failure processes in commercially available monolithic structural ceramics at elevated temperatures under sustained or cyclic loads is not well-developed. In a recent review,[19] a committee of experts cited this lack of understanding and the virtually nonexistent data base of creep-rupture or cyclic fatigue results for these materials. For composites, the understanding of ceramic-matrix behavior must be augmented with an understanding of the behavior of the fiber and the fiber/matrix interface.

6.1.1 Matrix Cracking

In extensive room-temperature testing of unidirectional reinforced glass- and ceramic-matrix composites, it is generally observed that matrix cracking is the first event in the failure process. While such cracking does not generally result in immediate catastrophic structural

failure in the composite materials of interest, excessive permeability, leakage, fiber corrosion, and mechanical property losses can result.

In monolithic ceramics, failure behavior can be described by fracture mechanics crack-growth models with due consideration given to the effects of microstructure and residual stress.[20] The composite cracking behavior is complicated by the presence of fibers that can impede crack propagation. The extent to which the fibers impede crack growth is expected to be influenced by fiber size, fiber content, and fiber-matrix interface characteristics.[15]

6.1.2 Time-Dependent Behavior

In monolithic ceramics at high stress levels, short-term failures occur by subcritical crack growth from pre-existing flaws. At lower stress levels and high temperatures, creep processes that involve the generation of new flaws[20] occur. Other processes appear to involve the blunting or healing of flaws.

The monolithic ceramic damage processes are expected to occur in the ceramic matrix of the composite material. Because the matrix in a ceramic composite is a significant load-carrying element, these processes are expected to influence the distribution of stress and the resulting failure mechanisms. A fundamental understanding of damage processes under temperature and environmental conditions is needed.

6.1.3 Fatigue

Load cycling tests[15] to stress levels that produce matrix cracking were observed to result in changes in elastic properties, suggesting nonrecoverable damage processes. Insufficient cyclic testing of ceramic composites to actual working stress levels has been done to determine the nature and effect of fatigue damage processes on long-term performance.

6.1.4 Ultimate Strength and Toughness

Ceramic (brittle matrix) composites tend not to behave as linear elastic materials at high stress. To assess the capability of a heat

exchange component to survive upset load conditions, the (damage) processes that affect nonlinear behavior need to be understood. It is believed that the fiber-matrix interface is an important aspect controlling ultimate strength and toughness.[21]

6.1.5 Test Techniques

Ceramic-fiber, ceramic-matrix composites constitute a relatively new class of materials with considerable promise for solving problems associated with monolithic ceramics. For the composite system of choice, very extensive mechanical testing of unidirectionally reinforced material and simple laminates (or other fiber geometries) will be required to develop an understanding of the failure process(es). This testing must include extensive microstructural examination of stressed and failed specimens to establish the sequence of events (matrix cracking, fiber debonding, fiber fracture, etc.) leading to ultimate failure. The application of micromechanical failure models[15,11] can serve as a guide for the testing and examination. Generation of the required design data is difficult because many of the material systems are not mature and because satisfactory testing techniques have not been fully developed. An evaluation of test methods for ceramic-matrix composites is given in Ref. 22.

The test methods can parallel those developed for brittle polymer-matrix composites[19] with the added dimension of high-temperature testing. Test methods used for metal-matrix composites are also useful in designing specimens for determination of off -axis and shear properties, and for multiaxial stress states.[23,24] The unique aspects of ceramic-matrix composites that require modification of testing techniques used for other materials include ratios of fiber modulus to matrix modulus near one, matrix cracking at lower stresses than fiber fracture, weak or unbonded interfaces, dramatic differences in elastic moduli in tension and compression, and much higher strength in compression than tension.

Designers typically require tensile, compressive, and shear data at the service temperature. Gripping ceramics and ceramic composites for mechanical testing is a difficult problem. The grips generally apply unwanted localized stresses and cause premature failure. Testing at

elevated temperature compounds the gripping problem. Nevertheless, progress is being made toward solution of this problem.[25]

6.2 RELIABILITY

The scatter in failure data of monolithic ceramics has been attributed to inherent flaw populations and low fracture toughness. Significant scatter in organic-matrix composites has also been observed and studied.[17] Variations in fiber volume fraction, fiber alignment, and individual fiber strengths are thought to contribute to this variability.

It is believed that significant scatter in matrix and fiber behavior will influence ceramic composite behavior. Because material behavior and damage processes for these materials are not well understood, it is anticipated that deterministic approaches will precede models that account for the inherent scatter in these materials.

6.2.1 Phenomenological Design Limits

Stress or strain limits that depend upon operating temperatures and environments, must be established to ensure that functional requirements are satisfied throughout the useful life of ceramic composite components. These limits need to encompass the conditions under which damage mechanisms are expected to lead to loss of function.

Several mechanisms must be addressed in establishing design limits: matrix cracking, prolonged high-temperature and environmental exposure effects, creep, residual stresses, and load-cycling effects. These issues may influence elastic stiffness properties as well as strength properties, thereby causing a redistribution of internal stress and a resulting effect on damage rates.

Continuum models are traditionally used to describe design limits. These models are based on strength of materials and fracture mechanics principles and often are derived from micromechanics considerations.

6.2.2 Acceptance Criteria

Physical characteristics like density, voids, flaws, inclusions, and surface finish can be related to the strength behavior of monolithic ceramics. While these characteristics are expected to have a somewhat different influence on composite ceramics, additional physical features like matrix-rich areas, fiber clumping, and fiber alignment can influence composite strength behavior. In addition, features of the fiber-matrix interface are expected to strongly influence ultimate strength and toughness properties.

A reliable design methodology should include an understanding of the detectable physical characteristics that significantly influence composite strength behavior. Traditionally, this understanding provides the basis for acceptance criteria compatible with phenomenological design limits.

6.2.3 Risk/Reliability Assessment

Both monolithic ceramics and traditional (organic) matrix composites have exhibited considerable scatter in strength properties. Design methodologies in monolithic ceramics were initially based on Weibull weakest-link methods[13] that have recently been further developed through the incorporation of fracture-mechanics concepts.[15,16]

The anticipated significant scatter in composite strength behavior suggests that further development of the weakest-link concepts to anisotropic ceramic composite materials will be needed.

7. STRUCTURAL VALIDATION

As previously indicated, tests of smaller-scale specimens with relatively simple geometries are typically employed to guide the development of analysis methods and design criteria. To confirm the validity of the overall design methodology, structural tests and analyses are required. Such tests are often designed to simulate, to a reasonable extent, certain features of the geometry and loading conditions of actual structural components. For example, actual fiber-reinforced ceramic heat-exchanger tubes might have a 2-in. diameter and a 30-ft length. Tests at elevated temperature, under axial load and internal pressure, could be conducted on tubular specimens with about the same diameter, wall thickness, and fiber architecture as the heat-exchanger tubes but with a 2-ft length. To assess potential modes of failure, stress levels would have to exceed those expected under normal operating conditions of the heat exchanger.

Temperature, pressure, load, and deformations should be accurately measured and controlled, and microscopic posttest examination of the specimens should be performed to obtain maximum benefit from such tests. Detailed structural analyses of the tests should also be performed. The validity of the analysis methods and the failure criteria can then be assessed on the basis of comparisons between the results of the tests, examinations, and analyses.

8. DEVELOPMENT AND VALIDATION NEEDS

The use of fiber-reinforced ceramics in advanced heat exchangers can lead to considerable economic benefits to U.S. industry. However, several significant development and validation needs must be satisfied to establish a viable design methodology for such materials. These needs are discussed below in the context of the key ingredients of the methodology.

The principal objective of the design-methodology development is to establish failure-prediction models based on known damage mechanisms for ceramic composites in industrial heat-exchange equipment applications. Specific goals should include the following:

1. Determine (experimentally) the mechanisms of failure under complex load, temperature, and environmental conditions.
2. Develop a fundamental understanding of the pertinent damage and failure mechanisms.
3. Assess the applicability of fracture mechanics to the modeling of failure.
4. Identify the pertinent mechanical-property tests and test methods needed to characterize material properties.
5. Identify the design stress or strain limits for which functional requirements will be reliably achieved throughout the life of a ceramic component.
6. Identify the detectable physical characteristics of the composite ceramic that most strongly influences damage and failure mechanisms.
7. Develop analysis methods to predict the distribution of stresses resulting from thermal and mechanical loads on the anisotropic ceramic composite material.
8. Develop and validate failure prediction models for both short- and long-term failure mechanisms.

The approach should include both experimental and modeling efforts. These efforts should be iterative, with initial experiments designed to provide insights pertinent to the modeling effort. Subsequent iterations should address verification and improvement of the analysis and

failure prediction models. Finally, mechanical-property testing and small-scale structural testing should be utilized to validate design methodology.

8.1 MATERIALS TESTING

Designers typically require tension, compression, and shear data at the service temperature. For anisotropic composites, property data are needed in the principal material directions relative to the fiber orientation.

8.1.1 Test Methods Development

Development of suitable test methods for characterizing properties at temperature is needed. Gripping ceramics and composites introduces localized stresses that can produce premature failure. Testing at elevated temperatures compounds the gripping problem.

8.1.2 Mechanical Properties Data Base

A life-prediction design methodology requires reliable data for validation and for design use. The extent to which the test methods just described can be used to validate design methodology should be evaluated by suitable testing of a candidate material system.

8.2 ANALYSIS METHODS

8.2.1 Constitutive Relationships

Several analysis methods have been and are being developed for specific composite material systems as described in Chap. 5. The choice of analysis approach is determined by the material, fabrication method, and intended service environment.

Regardless of approach, effective anisotropic constitutive relationships between components of stress and strain should be developed and validated by laboratory tests.

8.2.2 Damage Models

Nonrecoverable damage processes, like matrix cracking and creep, may necessitate the use of nonlinear methods to predict the redistribution of stress and strain with time. Analysis methods that include consideration of damage effects mechanistically or phenomenologically should be considered.

8.3 FAILURE MECHANISMS AND DESIGN LIMITS

Both short- and long-term failure mechanisms are important to a viable design methodology for ceramic composites. Mechanisms controlled by prolonged stress or cyclic stress can alter the constitutive relationships with time and can cause delayed failure.

The influence of stress magnitudes and cycles at elevated temperatures and other environmental conditions should be established as a basis for phenomenological design limits and life-prediction models. Damage rates under various stress conditions should be ascertained for use in life prediction.

8.4 STRUCTURAL VALIDATION

Small component structural tests should be used to validate the overall design methodology, including materials characterization, constitutive models, damage models, and failure mechanisms. Such tests should simulate to a reasonable extent features of the geometry, fabrication method, and loading conditions of actual structural components. Several tests should be used to validate the various short- and long-term failure mechanisms.

9. RECOMMENDED APPROACH TO SATISFYING MOST SIGNIFICANT NEEDS

To establish a practical basis for design and analysis of HiPHES components, design methodologies for the use of continuous-fiber ceramic-matrix composites need to be developed and validated. As the first step, candidate materials must be identified. Currently two ceramic-fiber, ceramic-matrix material systems are being considered in the HiPHES program. One consists of Dupont PRD-166 (Al_2O_3 and 15 to 25% ZrO_2) fiber in an alumina (Al_2O_3) – zirconia (ZrO_2) matrix. This sol-gel derived oxide composite is to be fabricated as heat exchanger tubing by filament winding, which would result in a laminated structure with 2-D reinforcement. The other candidate material consists of Nicalon (59% Si, 31% C, and 10% O) fiber in a silicon carbide (SiC) matrix, fabricated by chemical vapor infiltration (CVI) of a 3-D, braided preform. The SiC composite would also require a protective plasma-spray coating of Al_2O_3.

Realistic representation of the effective mechanical properties of the candidate fiber-reinforced ceramic materials is the next significant step toward development of the design methodology. This step will require micromechanical modeling and material testing efforts. The modeling must account for the characteristics of the fiber, the matrix, and especially of the fiber-matrix interface. Thus, the testing must include uniaxial experiments at room and elevated temperatures to provide basic material properties. In addition to short-term tensile testing, long-term creep-rupture and cyclic fatigue experiments should be conducted at the temperatures and in the environments of interest to the HiPHES program.

In parallel with characterization of the material through fundamental tests and micromechanical modeling, macromechanical modeling needs to be performed. Namely, the macromechanical constitutive relationship between stresses, strains, temperature, and time must be established to describe the mechanical behavior of the material. Results of the basic uniaxial tests should also be incorporated into a mechanics model to predict the response of a multidirectionally reinforced circular tube.

Design criteria need to be developed as a basis for establishing stress or strain limits to provide adequate reliability against expected mechanisms of failure. Therefore, an understanding of failure processes in the reference materials is required. Results of the uniaxial tests can be employed to examine the prevailing failure mechanisms and the evolution of damage within the ceramic composite materials.

It is also recommended that structural tests be performed to investigate the behavior of reinforced tubular specimens under combinations of tensile load and internal pressure or combined tension-torsion loading. Results of structural tests are required to evaluate and to validate analysis methods and design criteria. Initially the modeling and testing effort should, for the most part, concentrate on unidirectional fiber reinforcement and on simple cross-ply laminates; but some attention should also be given at an early stage to braided fiber architecture.

Other federally funded agencies are conducting studies of fiber-reinforced ceramic-matrix composites. The results of these studies can have generic or material- and process-specific applicability to the HiPHES program. A brief survey of the ceramic-composites research activities of the National Aeronautic and Space Administration (NASA), the Air Force, and several other organizations is provided in the next chapter.

10. RESEARCH ACTIVITIES OF SEVERAL OTHER FEDERALLY FUNDED AGENCIES IN THE AREA OF CERAMIC COMPOSITES

Other federally funded agencies conducting research activities in the area of fiber-reinforced ceramic-matrix composites have been surveyed. The results of this brief survey demonstrate that, at present, design of the material represents the main thrust of the effort.

Ceramic composite research at NASA is addressed within the framework of the Advanced High-Temperature Engine Materials Technology Program (HITEMP). The main goal of NASA's HITEMP program is to advance the state of the art in composite materials and structural analysis, to enable further development of civil-aircraft propulsion systems.[26] The effort focuses on research in metallic/intermetallic-, ceramic-, and polymeric-matrix composites, analytical modeling, and testing methods.

In the area of ceramic-matrix composites, the emphasis of the HITEMP program is on materials that could be used at temperatures of up to 1650°C (3000°F). The research efforts are aimed at developing structurally reliable ceramic composites reinforced by long or continuous ceramic fibers. Like monolithic ceramics, the fiber-reinforced ceramics have considerably lower densities, better oxidation resistance, and the potential for operating at significantly higher temperatures than superalloys. In contrast to monolithic ceramics, the fiber-reinforced ceramics display metallike deformation behavior, noncatastrophic failure, and strength properties that are less sensitive to processing and service-generated flaws.

At present an adequate knowledge base, required to create tough fiber-reinforced ceramics with environmental stability to temperatures surpassing the operating temperatures of superalloys, does not exist. Therefore the NASA efforts in ceramic-matrix composite research will evolve in two phases.[27] The objective of the current phase I is the identification of materials and processing approaches for reliable microstructures for the fiber-reinforced composites. Research focuses on the selection, development, and integration of the constituents of these microstructures, namely fibers, matrices, and interfaces. The goal of phase II is to form reliable macrostructures that will meet the

structural and environmental requirements of the application. Thus, the emphasis of phase II will be on fiber-architecture effects, fabricability approaches for complex shapes, process cost-effectiveness, and joining techniques for ceramic composite materials.

In the area of fiber development at NASA, the ongoing studies are aimed at the development of SiC fibers with optimum properties for ceramic-matrix reinforcement.[28] Significant results have been obtained regarding the high-temperature stability of the SCS-6 carbon-rich coating for the SiC fibers. Two important advantages of the SCS-6 coating were determined: (1) fiber tensile strength was doubled and (2) a weakly bonded fiber-matrix interface was produced, which is essential for tough ceramic-matrix composites. The fiber strengthening may be caused by carbon healing of SiC sheath surface flaws. The beneficial weakening of the interface may result from debonding and slippage within the carbon layers. However, it was observed that long-term exposure to oxygen at temperatures above 400°C results in rapid removal of the beneficial carbon. It was also observed that exposure to an inert gas environment at temperatures above 900°C can accelerate the degradation of the outer coating surface, thus leading to a significant decrease in oxidation resistance. Based on these results research efforts will focus on (1) optimizing the SCS-6 coating by increasing the thermal stability of the outermost SiC particulate layer and (2) developing new oxidation-resistant coatings that will be applicable to small-diameter SiC fibers and will retain the SCS-6 advantages.

The second area of fiber evaluation involved experimental investigation and modeling of fiber deformation and fracture as functions of time, temperature, and stress. Test results demonstrated that at temperatures below 800°C fibers deformed elastically, while at temperatures above 800°C additional anelastic (or recoverable) strains were observed. Axial creep strains observed were generally small (typically less than the elastic strain). For temperatures above 1100°C and durations of <1 h, an exponential creep law was proposed. Tensile stress-rupture results indicated that under environmental conditions that do not severely degrade fiber coating or surface, fiber tensile strength decreases with time and temperature. A simple equation was shown to

accurately predict fiber rupture strength for times of 0.1 to 1000 h and over the 800 to 1300°C temperature range.

In addition, different processes for producing SiC fibers are being investigated. Progress has been made in producing high-purity ceramic fiber materials of small (10 to 20 μm) diameter using chemical vapor deposition.[29] Crystalline SiC fibers with fine-grained structure can also be prepared by (1) melt-spinning various organosilicon polymers, (2) crosslinking, and (3) pyrolyzing to high temperature (~1800°C). Advances in using this process to produce continuous crystalline SiC fibers with improved mechanical and thermal-stability properties were reported.[30]

To promote an understanding of the microstructure, the effects of temperature, matrix density, and interfacial shear strength on the mechanical properties of a ceramic-matrix composite are being investigated.[31] The composite, consisting of aligned 142-μm diameter chemical-vapor-deposited SiC fibers in a relatively porous reaction-bonded silicon-nitride (RBSN) matrix, exhibits metallike stress-strain behavior, failure beyond matrix fracture, and improved structural properties when compared with unreinforced monolithic material of comparable density. However, because of the unusually high matrix porosity, initial matrix cracking occurred at low stress. The mechanical properties were also effected by long-term (100-h) exposure to an oxidizing environment in the 600 to 1000°C temperature range. Development of an improved material motivated a study of the effects of various factors on the mechanical properties.

Experimental results demonstrated that the as-fabricated composite properties were retained up to 1000°C. However, beyond 1000°C, the ultimate tensile strength decreased dramatically. It was also observed that the high-temperature densification by hot isostatic pressing caused a marked increase in the density, elastic modulus, and first-matrix-cracking stress, and a decrease in the ultimate tensile strength and strain capability beyond matrix fracture for the SiC/RBSN material. In addition, tests indicated that the first-matrix-cracking stress and the elastic modulus increased with increasing interfacial shear strength between fiber and matrix. To further improve the SiC/RBSN composite,

studies of nitriding kinetics and interface properties of this material were undertaken.[32]

Studies are under way to develop a SiC-fiber-reinforced SiC-matrix composite characterized by high strength, high toughness, and sufficient oxidation resistance for structural applications at temperatures above 1400°C.[33] Another approach to the development of ceramic composites is being explored through a polymer-derived ceramics program.[34] The main objective of the polymer-derived ceramics program is the development of new polymers suitable as precursors to ceramic fibers, matrices, and/or coatings. The research activities include the synthesis of improved silicon-based polymers, the characterization of the polymer-to-ceramic conversion process, and the establishment of techniques for composite and fiber processing.

The NASA efforts in ceramic-matrix composite material development are complemented by analytical modeling.[35] Analytical modeling activities center around micromechanics and fracture modeling. In micromechanics, the objective is to develop constituent-based models to describe the nonlinear thermomechanical behavior of the ceramic-matrix composites. Several approaches are being pursued: (1) extending existing polymeric-composite micromechanics models, (2) developing the boundary element method (BEM), and (3) incorporating probabilistic micromechanics. In fracture modeling, the goal is to advance the development of fast fracture and reliability models with an emphasis on ceramic-matrix composites. This effort is progressing along two directions: (1) extending existing models for monolithic ceramics and (2) developing a probabilistic model. Currently a literature survey of existing mathematical models and finite-element codes for woven-fiber-reinforced ceramic-matrix composites is being conducted. The use of existing models will aid in acquiring a physical understanding of the structural behavior of the ceramic-matrix composites. Then further directions for material development research can be suggested.

Experimental results reveal that failure of the fiber-reinforced ceramic-matrix composites is governed by various micromechanical processes such as matrix microcracking, slipping between matrix and fibers, fiber-matrix interfacial debonding, delamination, and fiber breakage. A

local-global model, combining micromechanical and macromechanical analyses, has been proposed.[36] The local-global model considers the vicinity of the crack tip a "process zone" that is embedded in an anisotropic continuum, representing the bulk composite. Various micromechanical failure phenomena are modelled within the "process zone", while the bulk composite is modeled with conventional finite elements. Another effort in the micromechanical analysis of ceramic composites is aimed at adaptation of the BEM to the thermal stress analysis of these materials.[37] The BEM has been developed such that individual fibers embedded in a 3-D ceramic matrix could be analyzed. The analysis is capable of accounting for high stress gradients. In the future it is intended to extend this method to nonlinear and dynamic loading.

The Air Force Office of Scientific Research (AFOSR) is pursuing studies in fiber-reinforced ceramic-matrix composites through a number of contracts with universities. These focus largely on the micromechanical aspects of the ceramic composites, such as properties of fiber-matrix interfaces, microcracking, and microfailure modeling.

The Materials Laboratory of the Air Force Wright Research & Development Center (WRDC) plans to conduct an extensive study of composites consisting of Nicalon fiber in matrices of glass (Corning 1723), glass-ceramic (Corning LAS), and SiC. All three material systems will be acquired with unidirectional reinforcement and as 0/90 laminates. In the case of the SiC matrix, a braided Nicalon reinforcement will also be studied. In addition, a High-Temperature Composites Research Group has been established at WRDC to conduct research in high-temperature brittle-matrix composites.[38] The current goal of this effort is to gain a fundamental understanding of the behavior of brittle-matrix composites with the focus on ceramic-matrix composites. Current work concentrates on (1) mechanical behavior of ceramic-matrix composites and (2) fundamentals of high-temperature composites (i.e., study of the factors that determine the choice of constituents, processing, time-dependent behavior, chemical interaction, and microstructural stability). The long-term goal of this research is to develop a reliable analytical model, which would require as input only basic properties of the

constituents and interface obtainable from simple experiments. The model then should be able to predict elastic and fracture behavior of the composite and further guide the design of the microstructure. At present the ability to develop a comprehensive model is limited by (1) a shortage of measurement techniques for interface properties and (2) a lack of understanding of composite failure processes. A large fraction of the current and future work is devoted to addressing these issues.

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