ABSTRACT

Structural ceramics have been under nearly continuous development for various heat engine applications since the early 1970's. These efforts have been sustained by the unique properties that ceramics offer in the areas of high-temperature strength, environmental resistance, and low density and the large benefits in system efficiency and performance that can result. But the promise of ceramics has not been realized because their brittle nature results in high sensitivity to microscopic flaws and catastrophic fracture behavior. This has translated into low reliability for ceramic components and thus limited application in engines. For structural ceramics to successfully make inroads into the terrestrial heat engine market requires further advances in low cost, net shape fabrication of higher reliability components, and improvements in properties such as toughness, strength, etc. These advances will lead to very limited use of ceramics in noncritical applications in aerospace engines. For critical aerospace applications, an additional requirement is that the components display markedly improved toughness and noncatastrophic or graceful fracture. Thus our major emphasis on fiber-reinforced ceramics.

The NASA Lewis Research Center's Ceramic Technology Program is focused on aerospace propulsion and power needs. Thus, emphasis is on high-temperature use of ceramics and on their structural and environmental durability and reliability. The program is interdisciplinary in nature with major emphasis on materials and processing, but with significant efforts in design methodology and life prediction. About thirty-five researchers in the Materials and Structures Divisions are involved in the project. Strong interactions and collaborations between materials efforts and NDE, corrosion, fracture, and design methodology exist.
Structural ceramics have been under nearly continuous development for various heat engine applications since the early 1970's. These efforts have been sustained by the unique properties that ceramics offer in the areas of high-temperature strength, environmental resistance, and low density and the large benefits in system efficiency and performance that can result. But the promise of ceramics has not been realized because their brittle nature results in high sensitivity to microscopic flaws and catastrophic fracture behavior. This has translated into low reliability for ceramic components and, therefore, limited application in engines.
The results of recent studies of ceramic applications in small aeropropulsion engines have revealed that substantial benefits are possible over current engine technology. Small gains can be obtained via improved aerodynamic and cycle efficiency. Much larger benefits are possible by going to a regenerated cycle or by going to an uncooled hot section. Both of these approaches require ceramics, i.e., a ceramic regenerator for weight considerations and ceramic hot-section components to overcome the need for hot-section component cooling.
For structural ceramics to successfully make inroads into the terrestrial heat engine market, further advances are necessary in net shape fabrication of components with greater reliability and lower cost. The cost constraint as well as technical constraints currently dictate use of monolithic or possibly particulate or whisker-toughened ceramics. Improvements in properties such as toughness, strength, lubricity, and durability may also be needed for specific applications. In addition, technology advances in life prediction and nondestructive evaluation are required. These advances in technology will lead to very limited use of ceramics in noncritical applications in aerospace engines. For critical aerospace applications, an additional requirement is that the components display markedly improved toughness and noncatastrophic or graceful fracture. Thus our major emphasis on fiber-reinforced ceramics.
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Two basic forms of reliability can be defined for ceramics. The first is a statistical reliability as illustrated in the figure. Ceramics typically display a broad distribution of strengths. In the inspection approach to reliability, we would separate unacceptable parts by NDE and proof testing and reject them. A more efficient and cost-effective approach lies in improved processing that increases strength and yields no defects.

We define the second form of reliability as functional reliability because it relates to how well a component performs its function during system assembly and service. Thus, factors such as fracture toughness, impact resistance, and failure mode (graceful versus catastrophic), which are governed by micro- and macrostructure, need to be considered. Specifically, particulate and whisker phases can improve fracture toughness and continuous fiber additions can also provide a noncatastrophic failure mechanism. This brings us into the realm of engineered microstructures, i.e., composites.

**Approaches to Ceramic Reliability**

"Inspect in" the quality

- Separate out the unacceptable parts by NDE and proof testing and reject them.

**Requirement**

- Inefficient
- Costly

**Quality Control Approach**

- Efficient
- Low cost
- Reliable

Improve the Process

- Eliminate - inclusions
  - voids & cracks
  - surface flaws

**Requirement**

- No rejects and improved strength

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Research is focused on SiC and Si$_3$N$_4$ because these materials offer the desired combination of high-temperature strength, thermal shock resistance, and environmental durability. We are concluding efforts on SiC and Si$_3$N$_4$ reliability improvement. Future efforts are being focused on determining the potential of these materials for use in the 1300 to 1600 °C range. This requires improvements in strength and toughness and an understanding of how these improvements translate into use potential. These efforts are synergistic with our effort in fiber-reinforced ceramics where much of our emphasis is on SiC and Si$_3$N$_4$ materials.
Some recent progress at the NASA Lewis Research Center in improving the strength of silicon carbide is illustrated. Materials fabricated by dry pressing or slurry pressing, followed by sintering at 2200 °C for 30 min, have four-point flexural strengths of about 345 and 414 MPa, respectively. Hot-isostatic pressing tantalum-encapsulated, green, slurry-pressed specimens at 1900 °C for 30 min improves strength to about 552 MPa while achieving the same density. This densification at a much lower temperature yields a much finer grain size and a shift in the strength-limiting flaw from internal defects, such as pores and agglomerates, to surface machining defects. Improvements are being sought to reduce sensitivity to surface flaws.
In the area of silicon nitride processing, an improved NASA 6Y (6 wt % Y2O3) sintered Si3N4 composition was realized by iterative utilization of conventional x-radiography to characterize structural (density) uniformity as affected by systematic changes in powder processing and sintering parameters. Four-point flexural strength was improved 56 percent and the standard deviation was reduced by more than a factor of three. Correlated with these improvements were improved microstructures and a change in critical flaw character.
NASA has supported major contract research efforts to improve the statistical reliability and strength of silicon nitride (Garrett Ceramic Components Division) and silicon carbide (Ford Motor Co.) via improved processing centered about injection molding. Both efforts have made good progress toward the goals of 100 percent improvement in Weibull modulus and 20 percent improvement in strength. The effort at Garrett is essentially complete. One Garrett accomplishment, as shown in the accompanying figure, was the development of material GN-10, which appears to have significantly advanced the state-of-the-art.

IMPROVEMENTS IN SINTERED SILICON NITRIDE

IMPROVEMENTS IN SINTERED SILICON NITRIDE

NEW MATERIAL GN-10

1986 STATE-OF-THE-ART

FLEXURE STRENGTH, MPa

1000
900
800
700
600
500

0 200 400 600 800 1000 1200 1400

TEMPERATURE, °C

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Improved ceramic strength, toughness, and reliability can be achieved by incorporating continuous ceramic fibers. This gives stress-strain behavior that mimics a metal and noncatastrophic or "graceful" failure. The penalty for doing this is greater fabrication difficulty. Also, available fibers for high-temperature (1400 °C) ceramic matrix composites are limited, and the proper fiber-matrix bond must be maintained in fabrication as well as during the life of the composite. Too strong a bond yields a loss in toughness and a reversion to monolithic ceramic behavior, while too weak a bond yields loss in stiffness, strength, and toughness.

**FIBER-REINFORCED CERAMICS APPROACH TO RELIABILITY**

INCORPORATE CONTINUOUS CERAMIC FILAMENTS HAVING GREATER STIFFNESS THAN MATRIX

- **ADVANTAGES**
  - IMPROVED TOUGHNESS IMPARTED BY CRACK DEFLECTION AND CRACK BRIDGING
  - INCREASED MODULUS AND STRESS TO FAILURE
  - "METAL LIKE" STRESS-STRAIN BEHAVIOR
  - GRACEFUL FAILURE

- **DISADVANTAGES**
  - PROCESSING MORE DIFFICULT
  - AVAILABLE FIBERS LIMITED
  - CONTROL OF FIBER-MATRIX BOND REQUIRED
Reinforcing with ceramic fibers having a modulus and ultimate strength greater than the monolithic ceramic used as the matrix material yields ceramic composites with greater stiffness and greater strength at first matrix cracking. If small diameter fibers are used, matrix crack propagation can be delayed by the bridging mechanism depicted in the figure insert. This results in matrix failure for the composite at a stress and strain level higher than for the monolithic ceramic. If the fiber-matrix interfacial bonding is optimum, matrix cracks propagate around the fibers and not through them. Once matrix cracks start to form, they occur at a regular spacing. The ceramic is then held together by the load-carrying capacity of the fibers until they begin to fracture in a statistical manner. The net result for a tough ceramic composite having an optimum bond between the matrix and fiber is that a metallike stress-strain curve is displayed with first-matrix cracking stress corresponding to the yield stress of metals and fiber fracture corresponding to the ultimate strength.
CURRENT NASA LEWIS MATERIALS EFFORTS
— Fiber-Reinforced Ceramics —

The focus of current NASA Lewis research in fiber-reinforced ceramics (FRC) is on the development of fabrication approaches that yield good matrix properties and can be carried out with minimal degradation of fiber strength. Four approaches that are being pursued are outlined. Extension of the capability of FRC via development of advanced fibers and fiber coatings is a second area of focus. The third area of focus is assessment of FRC capability to perform in applications such as NASP and rocket propulsion systems. These efforts thus focus on key issues associated with each application, such as process scale-up to enable component fabrication, compatibility with the environment, and resistance to thermal shock.

CURRENT NASA LEWIS MATERIALS EFFORTS
—FIBER-REINFORCED CERAMICS—

I. PROCESSING STUDIES
• OPTIMUM MATRIX PROPERTIES
• FIBER STRENGTH RETENTION

<table>
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<tr>
<th>PROCESS</th>
<th>REACTION</th>
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<tr>
<td>Si POWDER + HEAT + N2 GAS + Si3N4</td>
<td>STRONG MATRIX</td>
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<tr>
<td>SiC POLYMER + HEAT + SiC</td>
<td>LOW-COST PROCESSING</td>
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<td>C POLYMER + HEAT + Si GAS + SiC</td>
<td>TAILORABLE MATRIX</td>
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<td>Al2O3 SOL GEL + HEAT + Al2O3</td>
<td>OXIDATION RESISTANCE</td>
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II. ADVANCED FIBER STUDIES
• HIGH STRENGTH
• STRENGTH STABILITY TO > 1650 °C (3000 °F)
• ENVIRONMENTAL PROTECTION
• OPTIMUM FIBER-MATRIX BOND

III. FRC APPLICATIONS
• NASP
• ROCKET PROPULSION
The fabrication sequence, microstructure, and mechanical properties of the high strength and toughness SiC fiber-reinforced, reaction-bonded silicon nitride composite recently developed at NASA Lewis are summarized in this figure. Silicon and SiC fiber monotapes are interleaved and subjected to a mild hot-pressing step to burn out the binder and provide some green strength. The composite is then nitried to convert the silicon to Si$_3$N$_4$. The resultant composite microstructure contains high levels of porosity, particularly between fibers. In four-point flexural testing, the composite exhibits a first-matrix cracking strength comparable to typical monolithic RBSN even though the matrix density at 2.0 gm/cm$^3$ is far lower than that of monolithic RBSN. The ultimate strength of the composite is more than twice that of a typical RBSN at both 23 and 40 percent fiber loading.
Tensile stress-strain data and fracture behavior of 30 vol% SiC/RBSN composites are illustrated in this figure. An additional strain occurs after matrix fracture at about 0.12 percent strain. The stress at failure is much higher than for first matrix cracking. The fracture surface exhibits the moderate fiber pullout required for achieving a strong, tough ceramic matrix composite. It is expected that with the development of high strength, smaller diameter SiC fibers, the fracture properties of the SiC/RBSN will improve significantly.
Four-point bend strengths for SiC/RBSN at room temperature, 1200 °C (2200 °F) and 1400 °C (2550 °F) are compared with data for fully dense, hot-pressed Si₃N₄, reaction-bonded Si₃N₄, and SEP SiC/SiC composite (one-dimensional). At elevated temperature, 23 vol % SiC/RBSN is stronger than both monolithics and more than twice as strong as the SEP SiC/SiC composite.
An example of studies aimed at improved ceramic fibers can be found in a recent in-house study of post-processing of Nicalon SiC fibers. This research involved high-temperature/high-pressure treatments of Nicalon in an attempt to determine if the fiber properties could be improved or stabilized. Results are summarized in the graph. Treatment at 1360 atm in argon results in about a 300 °C increase in the maximum exposure temperature for onset of strength degradation. This effect is transitory in nature. Thus, exposure to high temperature at 1 atm after pressure treatment gives the same results as exposure of a nontreated fiber. However, if high-temperature exposure is necessary only for processing of the composite, the pressure treatment approach has significant merit.
CONCLUDING REMARKS

For ceramics to achieve their promise, reliable and economical fabrication processes must be developed for monolithic, whisker-toughened, and fiber-reinforced ceramics. In addition, a basic understanding of the materials science of ceramics is required to enable the development of the processing and of the design and life prediction methodologies that will enable them to be utilized.

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<th>MATERIAL CLASS</th>
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<tr>
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