
Advanced Composite Materials
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/tacm20

# Recent advances in oxide-oxide composite technology

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Version of record first published: 02 Apr 2012.

*Adv. Composite Mater.*, Vol. 8, No. 1, pp. 17–23 (1999)


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## 1. BACKGROUND

It is now well-documented that SiC and C-based ceramic matrix composites (CMCs) are susceptible to embrittlement at intermediate temperatures (700–900°C): the so called 'Pest Effect' (Fig. 1). It is caused by the ingress of moist air through cracks that form upon thermomechanical fatigue (TMF). The consequence is relatively short life above the matrix cracking stress, $\sigma_0$, when the application requires cycling through the pest temperature. There is also evidence that even when operating below $\sigma_0$, overloads can introduce damage that results in life-limiting pest failures. While these problems do not adversely affect performance in either short life application (e.g. in rocket engines) or in space, they substantially limit applications when long life is needed, as in aero and power turbines, etc. Accordingly, for these applications, all-oxide CMCs are being explored.

A substantial technology base generated on SiC-based CMCs provides general benchmarks for assessing the progress being made with oxide materials. The most notable are as follows:

(i) Themostructural robustness requires that the material be capable of sustaining considerable inelastic strain locally in the vicinity of strain concentrations, such as holes/notches. These strains redistribute stresses and contribute to desirable CMC notch performance, comparable to that for metals (and much superior to monolithic ceramics, Fig. 2). Inelastic strains in the range 0.5 to 1% achieve this objective (Fig. 2).

(ii) The inelastic strain is governed by a combination of matrix cracking and frictional slip at the fiber/matrix interfaces. The friction is, in turn, governed by the fiber roughness and the shear properties of the fiber coating.

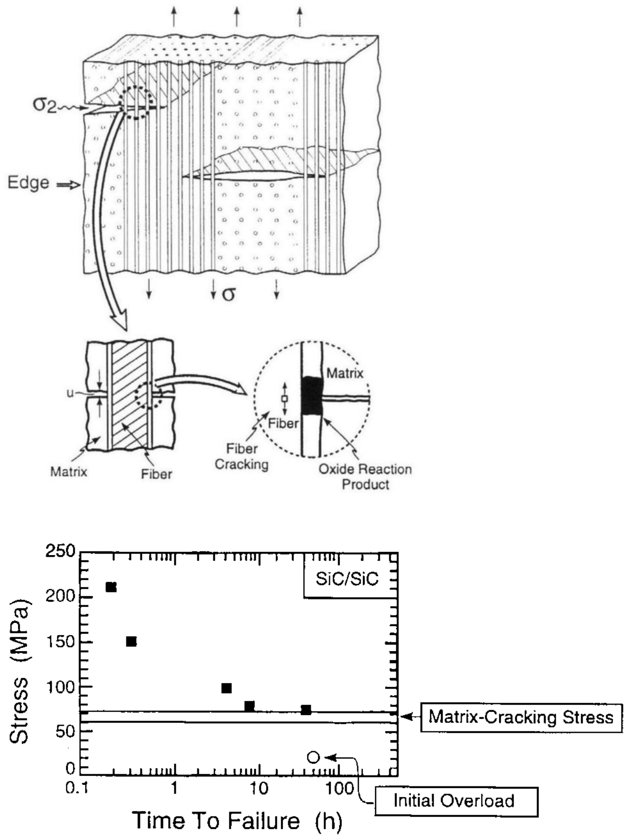


**Figure 1.** The oxidation embrittlement problem in SiC-based CMCs.

(iii) Absent environmental embrittlement, the fatigue threshold is a substantial fraction $\beta$ on the ultimate tensile strength (UTS): typically, $\beta = 0.7\text{–}0.8$. Fatigue above the threshold is attributed to changes in internal friction at the fiber/matrix interface.

(iv) The low interlaminar properties of 2D woven/laminated CMCs limit design flexibility and cause degradation both at attachments and in through-thickness thermal gradients.

## 2. OXIDE CMC MATERIALS

Two basic approaches have been used to create damage-tolerant, oxide CMCs. One uses a porous matrix with porosity designed to provide a compromise between in-plane damage tolerance and interlaminar strength. The other uses a monazite fiber coating which can be designed to give debonding with frictional slip at the fiber/matrix interface.

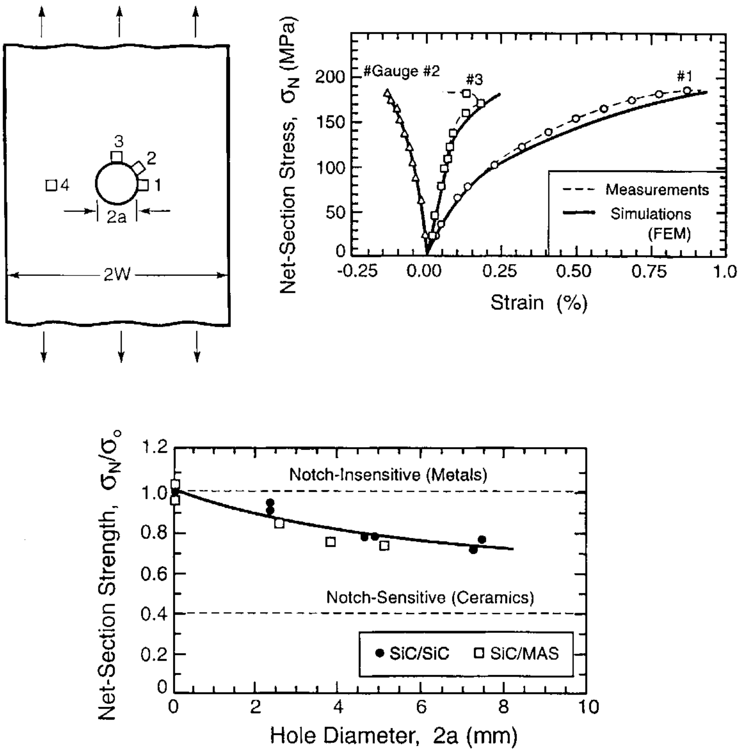


**Figure 2.** The inelastic deformation and notch tolerance of SiC-based CMCs.

### *2.1. Porous matrix materials*

The matrix microstructure has been designed to have a sufficiently low toughness to enable crack deflection through the matrix while maintaining enough strength for adequate off-axis and interlaminar properties. These seemingly contradictory requirements are achievable by incorporating a controlled amount of fine, uniformly distributed porosity. Acceptable matrix performance dictates a stable and well bonded particle network with substantial void space, ~30%, on a scale comparable with the interparticle spacing. Fine matrix particles are preferred to enhance packing density and uniformity within the fiber preform, as well as the nominal strength of the matrix. However, fine particles also reduce the stability of the matrix against densification during processing and service, promoting the formation of undesirable flaws under the constraint imposed by the surrounding fibers.

Mullite emerges as an attractive matrix material owing to its excellent creep resistance, low modulus and, sluggish sintering kinetics below ~ 1300°C. The latter suggests adequate microstructural stability for applications in the gas turbine engine, where initial target wall temperatures are in the range ~1000 to ~1200°C. However, it presents a challenge in processing. That is, temperatures above ~ 1300°C are required to achieve the requisite bonding between the matrix particles.

Yet most commercial oxide fibers are susceptible to microstructural degradation at these temperatures.

The matrix design concept is depicted in Fig. 3. Relatively large ($\sim$ 1 $\mu$m) mullite particles are packed between and within tows to form touching, non-shrinking network. Alumina particles that fit within the void spaces of this network ($\sim$ 200 nm) are added in a proportion limited primarily by the requisite levels of porosity. Since sub-micron alumina sinters readily above 800°C, the fine particles form bridges between the larger mullite particles, as well as between the mullite particles and the fibers, at processing temperatures which minimize fiber degradation. Interparticle voids may locally open owing to the sintering, but the overall matrix is constrained from shrinking by the rigid mullite network. The matrix is further strengthened by adding material to the alumina 'bridges' using precursor impregnation and pyrolysis.

The damage tolerance of these materials is manifest in their off-axis tensile stress/strain behavior and in the notch performance. That is, they exhibit considerable inelastic strain capability in the ±45° orientation (Fig. 3). Accordingly, even though they are essentially linear to failure in the 0/90° orientation, these materials exhibit high in-plane damage tolerance. In this regard they are 'fiber dominated' materials, analogous to C–C and polymer matrix composites.

These materials are limited in two ways. (i) They only retain long term damage tolerant behavior up to about 1200°C. At higher temperatures, sintering of the matrix to the fibers causes embrittlement. (ii) The transverse properties are marginal

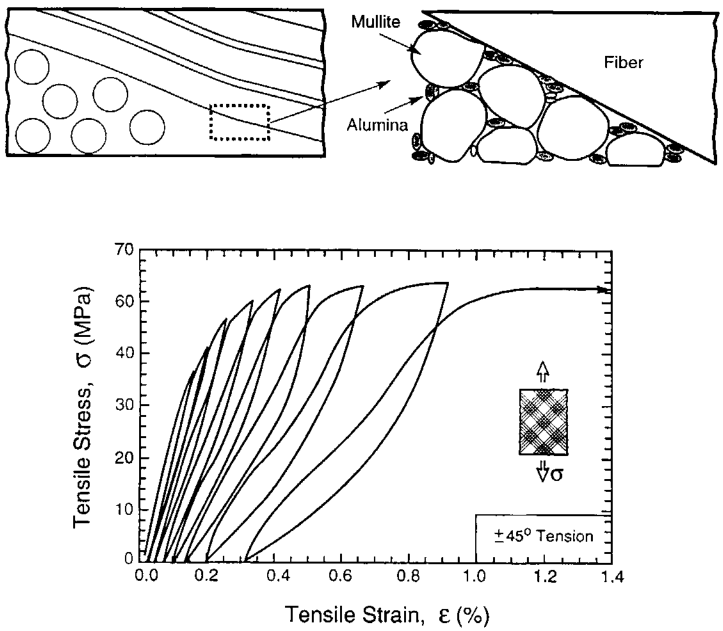


**Figure 3.** The characteristics of oxide CMCs with a porous matrix.

because of the matrix porosity. A 3D woven architecture is needed to achieve adequate design flexibility.

### *2.2. Monazite fiber coatings*

Monazite is thermochemically stable with $Al_2O_3$ and mullite and moreover, has a low interface fracture toughness. Accordingly, it has two of the three characteristics required from a fiber coating for an $Al_2O_3$ fiber-reinforced oxide CMC. By using a combination of mullite and monazite as a matrix, the compliance of the circumventing material provides the third characteristic; namely, the requisite frictional slip response between the fibers and matrix. All oxide CMCs of this type exhibit notch properties comparable to SiC-based CMCs (Fig. 4), while also achieving TMF longevity by eliminating the 'pest' degradation phenomenon. These are the most promising CMCs for long life application.

From an application perspective, oxide CMCs have the disadvantage relative to SiC materials that their thermal conductivity is appreciably lower. Accordingly, in high thermal flux conditions, the material temperatures are higher, placing greater demands on the creep and creep rupture performance. At this stage, these are the limiting properties of oxide CMCs. Efforts to increase the creep resistance by doping with rare earths (notably $Y_2O_3$) and by using nanoparticles are at the research forefront.

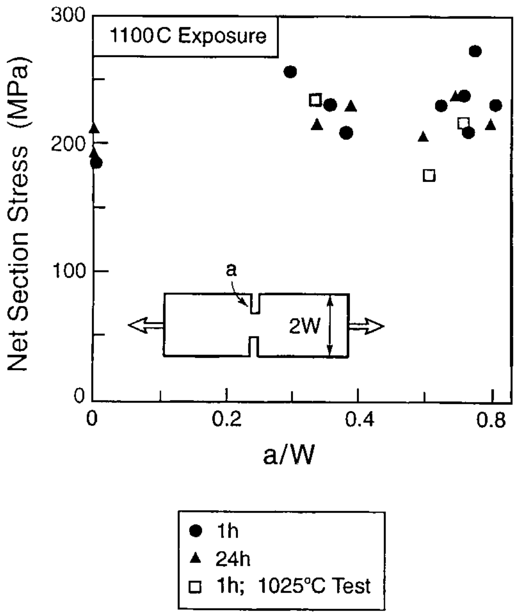


**Figure 4.** The performance of oxide CMCs with a monazite-based fiber/matrix interface.

## 3. INTERLAMINAR PERFORMANCE

The inferior interlaminar properties of 2D woven and laminated CMCs pose limitations on design flexibility and hence, on their implementation. The problems are manifest as delaminations that occur at thermomechanically loaded attachments as well as on plane sections subject to through thickness thermal gradients (Fig. 5). The interlaminar properties are controlled by the matrix material, which is designed to have low toughness to enable the fiber/matrix debonding and slip needed for in-plane damage tolerance. They are also subject to weakest link size scaling associated with manufacturing flaws, causing large components to be particularly susceptible to these problems.

The only solution appears to comprise 3D woven architectures: the same solution used for structural C–C composites. The weaving technology is under development for oxide fibers. Its viability has already been demonstrated.

## 4. SUMMARY

For applications requiring long life, oxide composites comprising $Al_2O_3$/mullite fibers with a monazite/mullite matrix have greatest applicability. Architectures

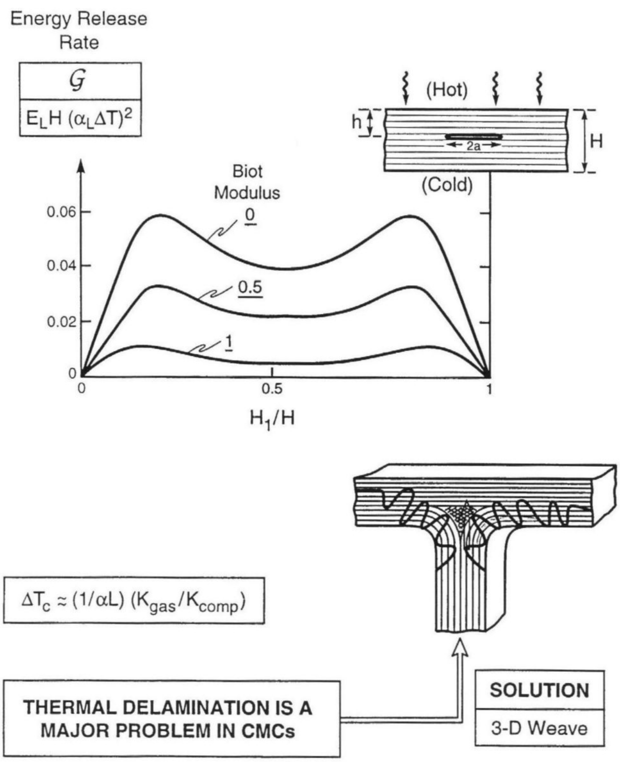


**Figure 5.** Delamination problems in 2D woven CMCs.

based on local 3D weaving are needed to obviate delamination sensitivity, particularly at joints/attachments and in regions subject to high thermal flux. These materials are presently creep/rupture limited. Research priorities include approaches for enhancing the creep resistance by using dopants or nanoparticles.

For either short life or space applications, C–SiC materials are often preferred because of their high-temperature capability at low oxygen pressures and their relatively high thermal conductivity.

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