Effects of Thermal Aging on the Mechanical Properties of a Porous-Matrix Ceramic Composite

Eric A. V. Carelli*
Science and Technology Center, Siemens-Westinghouse Power Corporation, Pittsburgh, Pennsylvania 15235

Hiroki Fujita,* James Y. Yang, and Frank W. Zok*
Materials Department, University of California, Santa Barbara, California 93106

The present article focuses on changes in the mechanical properties of an all-oxide fiber-reinforced composite following long-term exposure (1000 h) at temperatures of 1000–1200°C in air. The composite of interest derives its damage tolerance from a highly porous matrix, precluding the need for an interphase at the fiber–matrix boundary. The key issue involves the stability of the porosity against densification and the associated implications for long-term durability of the composite at elevated temperatures. For this purpose, comparisons are made in the tensile properties and fracture characteristics of a 2D woven fiber composite both along the fiber direction and at 45° to the fiber axes before and after the aging treatments. Additionally, changes in the state of the matrix are probed through measurements of matrix hardness by Vickers indentation and through the determination of the matrix Young’s modulus, using the measured composite moduli coupled with classical laminate theory. The study reveals that, despite evidence of some strengthening of the matrix and the fiber–matrix interfaces during aging, the key tensile properties in the 0°/90° orientation, including strength and failure strain, are unchanged. This strengthening is manifested to a more significant extent in the composite properties in the ±45° orientation, wherein the modulus and the tensile strength each exhibit a twofold increase after the 1200°C aging treatment. It also results in a change in the failure mechanism, from one involving predominantly matrix damage and interply delamination to one which is dominated by fiber fracture. Additionally, salient changes in the mechanical response beyond the maximum load suggest the existence of an optimum matrix strength at which the fracture energy in the ±45° orientation attains a maximum. The implications for long-term durability of this class of composite are discussed.

I. Introduction

The power generation industry has been under increased pressure to reduce NOx emissions from gas turbine engines while keeping up with market demands for increased power output and efficiency. These goals can be achieved in part through reductions in the amount of film cooling of combustor liners and turbine airfoils with attendant increases in the temperatures both within the gas turbine and at the burner outlet.1,2 In current gas turbine engines, many of the superalloy-based components are operating at or near their upper use temperature, even with the benefits imparted by the use of thermal barrier coatings, thereby precluding significant temperature elevations with these alloys. To meet future environmental and performance standards, it is anticipated that the targeted temperature elevations in turbine components will be accomplished through the use of continuous-fiber-reinforced ceramic composites (CFCCs). Among the various ceramic composites that have been developed to date, the ones that have attracted the greatest attention within the power generation industry in the past few years are those made from all-oxide constituents. The main advantage of the oxide-based composites over non-oxide ones (e.g., SiC/SiC) is their superior resistance to oxidation under typical turbine engine conditions and hence their potential for long-term durability. As part of a broad activity aimed at developing and assessing oxide composites for use in future generations of gas turbine engines, the present study focuses on changes in the mechanical properties of a candidate all-oxide CFCC following long-term exposure (1000 h) at temperatures of 1000–1200°C. The composite material of interest derives its damage tolerance from a highly porous matrix, precluding the need for an interphase at the fiber–matrix boundary. Although the efficacy of this material concept in enabling damage tolerance has been demonstrated,3–6 it remains to be established whether the matrix pore structure is stable against densification and whether the desirable damage-tolerant characteristics can be retained for extended time periods at the targeted service temperatures. Indeed, other CFCCs based on the porous-matrix concept have been shown to exhibit severe degradation in composite properties once the matrix densifies appreciably.7

The main matrix constituent in the present composite is mullite, in the form of a weakly bonded particulate network. This selection is based on the sluggish sintering kinetics of mullite8 at the upper use temperature for the fibers (1200°C for Nextel 720). This phase is intended to form a contiguous particulate network that should be immune from appreciable densification both during processing and under subsequent service conditions. The minor matrix constituent is alumina, present both in the form of particulates from a slurry and as a product of pyrolysis of an aqueous precursor solution.9 Because of its more rapid sintering kinetics, alumina serves to bond the mullite particulates and the fibers together, thereby enhancing the matrix-dominated composite properties, e.g., interlaminar strength and off-axis in-plane strength. However, if the degree of sintering becomes excessive, the damage-tolerant characteristics may be compromised. The challenge involves selection of the relative fractions and topologies of the two phases such that the network of mullite particles remains contiguous and hence prevents global shrinkage, yet the extent of bonding within this network is sufficient to impart the requisite matrix integrity for acceptable off-axis properties. These opposing requirements on the
matrix suggest an optimum state, dictated in part by the combination of properties that are required in the application of interest.

In this study, comparisons are made between the tensile properties of a 2D woven CFCC both along the fiber direction (0°/90°) and at 45° to the fiber axes before and after high-temperature aging treatments. These orientations are selected to elicit the fiber-dominated and matrix-dominated composite properties. Examinations of the broken specimens by optical and scanning electron microscopy are used to elucidate the role of aging in the fracture characteristics. Changes in the state of the matrix are probed through two additional complementary methods: (i) measurement of matrix hardness using Vickers indentation, and (ii) determination of the matrix Young’s modulus, using the measured composite moduli coupled with classical laminate theory. Additionally, some comparisons are made with the retention in properties of a comparable porous-matrix composite with an aluminosilicate matrix.

II. Materials and Test Procedures

The composite material consists of Nextel 720 fiber cloth in an 8-harness satin weave and a porous matrix of mullite and alumina. The matrix was produced in two steps. In the first, an aqueous slurry containing mullite and alumina particulates was vacuum-infiltrated into a stack of 12 fiber cloths. The matrix aqueous slurry containing mullite and alumina particulates was aged in the fracture characteristics. Changes in the state of the matrix are probed through two additional complementary methods: (i) measurement of matrix hardness using Vickers indentation, and (ii) determination of the matrix Young’s modulus, using the measured composite moduli coupled with classical laminate theory. Additionally, some comparisons are made with the retention in properties of a comparable porous-matrix composite with an aluminosilicate matrix.

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Table I. Summary of Physical Properties of CFCC Panels

<table>
<thead>
<tr>
<th>Panel designation</th>
<th>Fiber orientation</th>
<th>Matrix porosity (%)</th>
<th>Fiber volume fraction (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Initial†</td>
<td>Final‡</td>
</tr>
<tr>
<td>A</td>
<td>0°/90°</td>
<td>39.5</td>
<td>37.9</td>
</tr>
<tr>
<td>B</td>
<td>0°/90°</td>
<td>40.1</td>
<td>38.3</td>
</tr>
<tr>
<td>C</td>
<td>±45°</td>
<td>40.2</td>
<td>37.7</td>
</tr>
</tbody>
</table>

† After slurry infiltration and drying. ‡ After precursor impregnation and pyrolysis and final sintering treatment.

III. Experimental Results and Analysis

Typical micrographs of polished cross sections of both an as-processed specimen and one aged at 1200°C are shown in Fig. 1. A notable feature is the presence of a more-or-less regular pattern of matrix cracks, caused by the constrained shrinkage of the matrix during drying of the green panels. In the as-processed composite, the cracks are concentrated in the matrix-rich regions.

Fig. 1. SEM micrographs of as-processed and thermally aged composite specimens (viewed in backscatter imaging mode).
between fiber tows. They arrest at the interface with the longitudinal fibers (oriented perpendicular to the crack plane) and penetrate only slightly into the transverse tows. Following aging, the pattern of matrix cracks remains essentially the same, with the exception that the cracks tend to grow into the transverse tows and their opening displacement increases somewhat (see, for example, the cracks on the right side of Fig. 1(b)). These features are believed to be due to some matrix densification in the matrix segments contained between the cracks along the direction perpendicular to the cracks. Since this shrinkage is constrained by the (dense) fibers in the adjacent longitudinal tows, the shrinkage strain is accommodated by additional opening displacement of the cracks such that the net average strain is approximately zero. This hypothesis is supported by the measurements of the composite porosity, which indicated no significant change after any of the aging treatments. Higher-magnification SEM examinations of the matrix microstructure did not reveal any other obvious changes due to aging.

Representative stress–strain curves for the as-processed and the aged specimens in the 0°/90° orientation are plotted in Fig. 2(a). The variations in the Young’s modulus, \( E \), and the ultimate tensile strength, \( \sigma_u \), with aging temperature are summarized in Figs. 2(b) and (c). The only significant change is the slight increase in the modulus, from \( \approx 60 \) GPa in the as-processed condition to \( \approx 70 \) GPa after the 1200°C aging treatment. The tensile strength and the failure strain, \( \varepsilon_f \), remained unchanged; the averages and standard
deviations from 14 tests are $\sigma_e = 145 \pm 8$ MPa and $\varepsilon_e = 0.26\% \pm 0.03\%$. Also shown for comparison in Fig. 2(c) is the retained strength of a comparable all-oxide ceramic composite consisting of the same Nextel 720 fibers in an aluminosilicate matrix (in place of the mullite/alumina matrix used in the present study), after the same aging treatments.\(^7\) The strength of the aluminosilicate-based material decreases rapidly for aging temperatures beyond 1000°C, reportedly due to extensive densification of the matrix and an attendant loss in damage tolerance.

In the $0^\circ/90^\circ$ as-processed tensile specimens, the locations of the tow failures were uncorrelated with one another, as evident in the macrophotograph in Fig. 3(a). Indeed, the tow failure sites were offset by distances up to several centimeters along the loading direction. Similarly, highly uncorrelated fiber fractures were obtained within each longitudinal tow. An example of a broken tow near the fracture surface is shown in Fig. 4(a). A particularly striking feature is the seemingly large lateral separation between adjacent fibers. This feature is somewhat misleading in the sense that there are large longitudinal separations between the fiber fracture sites and hence many of the broken fibers within a broken tow are well outside the field of view when imaging the tow at even modest magnifications. These observations attest to the efficacy of the matrix in mitigating stress concentrations around fiber breaks and hence yielding damage tolerant behavior. Higher-magnification SEM observations revealed only small amounts of matrix particulates remaining adhered to the fiber surface (Fig. 4(b)). This result suggests that failure involves debonding and sliding either at or very near the fiber–matrix interface during fiber fracture, analogous to that in dense-matrix CFCCs with weak interphases. Similar features were observed on the specimens that had been aged at either 1000°C or 1100°C (e.g., Fig. 3(b)).

Following aging at 1200°C, the fiber tow failures in the $0^\circ/90^\circ$ specimens remained largely uncorrelated with one another at the macroscopic level (Fig. 3(c)). However, there was a noticeable increase in the correlation in the fiber failure sites within each tow (Fig. 4(c)). The most highly correlated failure sites appeared in small clusters, each containing perhaps 3–10 fibers. Additionally, the amount of matrix material remaining adhered to the fiber surfaces was significantly greater than that in the as-processed material. These features clearly indicate that both the matrix and the fiber–matrix interface have been strengthened as a consequence of the aging treatment, thereby reducing somewhat the extent of damage tolerance. Nevertheless, the effects do not appear to be sufficiently large to noticeably alter the $0^\circ/90^\circ$ composite strength.

In the $\pm 45^\circ$ orientation, the effects of matrix strengthening on aging were more pronounced (Fig. 5). In all cases, the tensile response was characterized by elastic–plastic behavior, reminiscent of metal plasticity (albeit at lower levels of strain). The transition from elastic to plastic behavior was gradual and the ultimate tensile strength was controlled by a plastic instability analogous to necking in metals,\(^4\) at an average strain of 0.32 ± 0.03%, independent of aging treatment. By contrast, Young’s modulus and the tensile strength increased dramatically following aging, by as much as a factor of 2 at the highest aging temperature. This trend reaffirms that some strengthening of both the matrix and the fiber–matrix interface occurs during aging. In the context of off-axis composite strength, such changes may be beneficial.

In the as-processed $\pm 45^\circ$ tensile specimens and the ones aged at temperatures up to 1100°C, failure occurred mainly through the matrix and was accompanied by extensive interply delamination and fiber “scissoring,” but with minimal fiber fracture (Figs. 6(a) and (b)). A consequence of this “scissoring” is through-thickness swelling in the region near the fracture surface. Following the
1200°C aging, there was a reduction in the extent of delamination. Furthermore, the failure mechanism now included extensive fiber fracture (Fig. 6(c)). This feature is consistent with the increases in the matrix and interface strengths due to aging, which improve the effectiveness of load transfer from the matrix to the fibers and hence increase the propensity for fiber fracture.

When viewed in the SEM (Fig. 7(a,b)), the fracture surfaces of the ±45° as-processed specimens revealed somewhat greater amounts of matrix particulates adhered to the fiber surfaces than that in the 0°/90° orientation. It is surmised that these differences are related to the differences in the stress states at the fiber–matrix interfaces in the two orientations, coupled with differences in the amounts of sliding that occur between adjacent fibers. The fracture surfaces of the specimens aged at 1200°C exhibited similar features but with greater amounts of matrix on the fibers, consistent with the trend seen in the 0°/90° orientation.

Salient changes in the fracture properties in the ±45° orientation are revealed in the tensile response following strain localization (beyond the load maximum). The load–displacement response in this regime (Fig. 5(c)) can be viewed as the traction law that would be pertinent to the fracture process zone in a notched specimen and hence provides information about the steady-state fracture energy. The results indicate that, as the matrix strength is initially increased, e.g., from the as-processed condition to the one obtained after 1000 h at 1100°C, the stress–displacement response is simply shifted up to higher strength levels, commensurate with the increase in the ultimate tensile strength. The fracture energy increases proportionately, by ~25%. However, as the matrix strength is increased further, e.g., after the 1200°C aging, the stress–displacement response begins at a higher
stress level but rapidly diminishes with increasing displacement, with complete rupture ensuing at a relatively small displacement. The corresponding fracture energy is reduced significantly, by more than a factor of 2 relative to that after the 1100°C heat treatment. This reduction in fracture energy can be attributed to the transition in the fracture mechanisms, from one of matrix cracking, delamination and fiber scissoring, to one involving extensive fiber fracture. This trend suggests the existence of an optimum matrix condition (e.g., strength) at which the fracture energy in the ±45° orientation attains a maximum.

The changes in matrix hardness with aging temperature are plotted in Fig. 8(a). The hardness was essentially constant (≈50 kg/mm²) up to 1100°C. It subsequently increased and reached a value of ≈100 kg/mm² following the 1200°C aging. This twofold increase in hardness is consistent with the twofold increase in the composite tensile strength in the ±45° orientation following the same aging treatment. The corresponding changes in the matrix Young’s modulus are plotted in Fig. 8(b). In light of the presence of the processing-induced matrix cracks (Fig. 1), this modulus represents an average value that incorporates the effects of the cracks and is therefore expected to be somewhat lower than that of the porous matrix itself. Nevertheless, since the density of cracks does not change appreciably during aging, the relative changes in the inferred matrix modulus are expected to reflect the changes due to matrix sintering. For the pristine composite, the inferred matrix modulus is in the range ≈5–9 GPa (more than an order of magnitude lower than that of fully dense mullite). Furthermore, the value inferred from the ±45° tensile tests is consistently higher than that from the 0°/90° tests. It is surmised that this difference is due to the different effects of the processing-induced matrix cracks (Fig. 1) on the average matrix modulus in the two testing orientations. The modulus increased with increasing aging temperature, especially above about 1100°C, and essentially doubled after the 1200°C aging. These results reveal yet again that changes occur in the state of the matrix as a result of the aging treatment.

IV. Discussion and Conclusions

The mullite/alumina matrix undergoes some degree of sintering during the aging treatments. The main manifestations are increases in modulus and hardness, by as much as a factor of 2. These changes cause similar elevations in the modulus and the tensile strength of the composite in the ±45° orientation, but with no change in the failure strain. Additionally, there is a noticeable increase in the propensity for fiber fracture in this orientation after the highest temperature aging treatment and evidence of reduced damage tolerance in the post-load-maximum regime. By contrast, in the 0°/90° orientation, the composite modulus increases only slightly and the tensile strength and the failure strain remain unchanged. Perhaps the most notable change in the latter orientation is the increase in the spatial correlation in the fiber failure sites within an individual tow and the increased amount of matrix material adhered to the fibers. Nevertheless, the failure sites appear to be sufficiently decorrelated from one another to suggest that the matrix largely continues to serve its role of mitigating stress concentrations around fiber breaks. In light of the known sintering kinetics of mullite and alumina, it is surmised that the sintering within the matrix is associated predominantly with the Al₂O₃, both from the particulates and that derived from the precursor.

The rather large differences in the effects of the matrix changes on the 0°/90° and ±45° composite properties can be rationalized with the aid of the schematic in Fig. 9. Since the properties in the ±45° orientation are dominated by the matrix, it follows that changes in the matrix properties will be translated in a roughly proportionate amount in the composite properties. This behavior is
the matrix modulus in the as-processed condition. Notably, the extent of matrix sintering in the aluminosilicate matrix CFCCs is much larger than that observed in dense-matrix CFCCs with weak interphases. This suggests the existence of a rather broad maximum or plateau in the fiber bundle strength, wherein the properties are insensitive to the matrix properties (top curve in Fig. 9). The much larger changes in the matrix strength following aging are illustrated by the decreasing part of the top curve in Fig. 9.

It is anticipated that there are two other behavioral regimes with regard to the 0°/90° and ±45° tensile response. In the former orientation, the strength is expected to increase with increasing matrix strength in the regime where the matrix strength is very low. This expectation is based in part on analogous behavior of dense-matrix CFCCs with weak interphases. Notably, when the interfacial sliding stress, \( \tau_{0} \), is sufficiently low to ensure global load sharing characteristics, the bundle strength is predicted to scale with \( \tau_{0}^{1/(m+1)} \) where \( m \) is the Weibull modulus of the fibers. For typical values of \( m \) (≈4–10), the exponent \( 1/(m + 1) \approx 0.1–0.2 \), and thus the sensitivity to \( \tau_{0} \) is weak. Indeed, over the typical range of \( m \) values, twofold changes in \( \tau_{0} \) only alter the bundle strength by \( \approx 10\% \), an effect which may not be detected readily among the scatter in the experimental measurements. Furthermore, once \( \tau_{0} \) becomes sufficiently large, the load-sharing characteristics among broken fibers become more localized and the fiber bundle strength then gradually diminishes. By analogy, the fiber bundle strength in the porous-matrix composites is expected to follow a similar dependence on the matrix shear strength, initially increasing and then decreasing with increasing matrix strength. Furthermore, the seeming independence of the composite strength on the matrix strength in the present experiments may be a consequence of a similarly weak dependence in composite properties on matrix properties in the regime probed by these experiments, coupled with some scatter in the experimental measurements. Consequently, it remains to be established more definitively whether the present experimental results do indeed reside along the broad plateau in Fig. 9 or whether they are in one of the adjacent regimes in which the strength is either gradually increasing or gradually decreasing with the degree of matrix sintering.

For similar reasons, a maximum in the ±45° composite strength is also expected. That is, once the matrix strength achieves a sufficiently high value, the inelastic straining capabilities of the composite are diminished and the strength of the composite becomes increasingly sensitive to the presence of flaws, introduced either during processing or as a consequence of mechanical loading. In this regime, the strength is expected to be low and exhibit large variability. These hypotheses require further theoretical and experimental investigation.

From a technological viewpoint, the retention of the fiber-dominated properties after the 1200°C aging treatment is particularly encouraging. It reaffirms that, with the selection of a matrix with a stable pore structure coupled with a stable oxide fiber, these composites have the potential for long-term durability at temperatures to which they will be subjected in the targeted applications. Otherwise, if the matrix is susceptible to appreciable sintering, the composites are prone to severe property degradation. Moreover, it is anticipated that further enhancement in the stability of the pore structure could be achieved through modifications to the matrix formulation, e.g., increasing the ratio of mullite to alumina, to ensure that the properties are retained for even longer time periods than those probed by the present experiments. However, these improvements may come at the expense of reduced off-axis properties. This is the subject of current investigation.

**Appendix**

Young’s modulus of the matrix was inferred from the measured Young’s moduli of the composites, measured both in the 0°/90° and ±45° orientations, using classical laminate theory. For this purpose, the composite is treated as a balanced, symmetric lay-up of unidirectional fiber composite plies. The calculation proceeds in two steps. In the first, the properties of the laminate are expressed illustrated by the lower (solid) curve in Fig. 9. In the 0°/90° orientation, the properties are largely fiber-dominated, with only small contributions coming from the matrix. For instance, because of the extremely low value of matrix modulus in the as-processed composite, even a twofold increase in this modulus following aging has only a small effect on the composite modulus. The main role of the matrix in this orientation is to act as a medium for blunting cracks that emanate from fiber breaks. Evidently the extent of the changes in the matrix strength following aging are insufficient to noticeably alter the fiber bundle strength. This suggests the existence of a rather broad maximum or plateau in the fiber bundle strength, wherein the properties are insensitive to the matrix properties (top curve in Fig. 9). The much larger changes in the extent of matrix sintering in aluminosilicate matrix CFCCs lead to extremely brittle fracture characteristics and low strength, illustrated by the decreasing part of the top curve in Fig. 9.

![Fig. 8.](image1.png) Effects of thermal aging on (a) the matrix hardness and (b) the matrix Young’s modulus.

![Fig. 9.](image2.png) Schematic showing the trends in the ±45° and 0°/90° tensile strengths with the degree of matrix sintering.
The fitting parameters, $\xi_i$ in Eqs. (A-3) and (A-4) are taken to be $\xi_G = 1$ and $\xi_k = 2,8,11$.

The matrix modulus was inferred from the measured modulus $E_{9090}$ via Eqs. (A-1) and (A-3), along with the known constituent properties ($E_t = 260$ GPa),$^{12}$ $v_m = v_f \approx v_{12} \approx 0.2$ (Ref. 13). Similarly, it was inferred from the measured modulus $E_{45}$ via Eqs. (A-2) and (A-4) and the same constituent properties. The results of these calculations are plotted in Fig. 7(b).

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References