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TENSILE AND FATIGUE BEHAVIOR OF A SiC/SiC COMPOSITE AT 1300°C


ABSTRACT: Monotonic and fatigue properties of a SiC/SiC composite were studied at 1300°C in tension. All tests were conducted in nitrogen (N₂) to eliminate oxidation. Because of the composite architecture and the differences between matrix and fiber properties, the composite failure under both monotonic and cyclic loads took place in stages. In a monotonic test, the composite failure occurred by the creep of bridging fibers, and thus, fibers displayed nonbrittle fracture features. In fatigue tests, although creep was also occurring under cyclic loads, the fracture surface analysis showed that final fracture took place in a brittle manner.

KEYWORDS: Silicon carbide, creep, fatigue, high temperature, tensile

INTRODUCTION

Silicon carbide (SiC) based ceramics are receiving much attention for their potential in high temperature applications. Although they have very good physical and chemical properties their use in monolithic form is limited due to their brittle nature. To solve this problem the fibers are commonly incorporated into the SiC matrix. The continuous SiC fiber-reinforced SiC composites synthesized by chemical vapor infiltration (CVI) are particularly attractive since this combination leads to a composite with very high fracture toughness [1].

As high temperature structural materials, the SiC/SiC composites are expected to be used under both static and cyclic loads. Therefore, the investigation of their response under these conditions is an important area of study for their proper use. As compared to room temperature tests, however, high temperature studies on composites are much more difficult since there are numerous experimental variables which need to be considered. Furthermore, since composites typically have at least two constituents with different properties, the response of each constituent becomes important while selecting a proper test

1 Associate Ceramist, 207 Metals Development, Ames Laboratory, Metallurgy and Ceramics Program, Ames, IA 50011.
condition. In this particular SiC/SiC composite, for instance, the oxidation of the carbon layer at fiber/matrix takes place readily below 1000°C [2]. The loss of the interfacial carbon layer is a very serious problem in this material since the mechanical properties deteriorate as a result [3,4]. Without some overlay coating it is almost impossible to study the long term response of this composite at elevated temperatures. Even then, as the matrix cracks develop, the oxidation of carbon occurs easily. Thus, the purpose of this investigation was to eliminate the oxidation using an inert atmosphere and to study tensile and fatigue behavior of a SiC/SiC composite at 1300°C.

EXPERIMENTAL PROCEDURE

Test Specimen

A commercial SiC/SiC composite with Nicalon fibers in 0°/90° orientation was obtained from Du Pont Lanxide Composites, Inc. (Newark, DE, USA). Fiber bundles for the composites were first coated with 0.5 μm pyrolytic carbon and then the CVI method was used to form the SiC matrix around the 2D laminates. The composite with 3.3 mm thickness had about 40% fiber volume and about 10% porosity. The uniaxial tensile tests were carried out with dog-bone shaped specimens having a 150 mm overall length and a 28 mm gauge length (Fig. 1). The width of the test specimens was about 12 mm and 10 mm at the end and gauge sections, respectively. To protect hydraulic wedge-grips from abrasion during clamping, copper plates were glued onto the specimen ends.

![Fig. 1. SiC/SiC composite tensile specimen used in this study.](image)

Mechanical Testing

Mechanical tests were conducted in a servo-hydraulic unit equipped with water-cooled hydraulic grips, a short furnace with graphite heating elements capable of reaching 2000°C and a high temperature contact extensometer with 25 mm gauge-length. As shown by the sketch in Fig. 2, the extensometer was positioned outside of the hot-zone, and the strain was measured by means of the SiC contact rods. The loading apparatus
shown in the sketch was contained entirely in an environmental chamber to carry out tests in controlled atmospheres. Since the ceramic-based materials are brittle and generally exhibit very small deformation, the alignment and the stability of the whole unit at all temperatures have been found to be extremely critical for the acquisition of accurate and reproducible data. Prior to tests the load-train was carefully aligned in order to minimize the bending moments, and thus to prevent premature failure. This was accomplished by means of a dummy specimen containing twelve strain-gauges. At the end, a bending strain of less than 3% was obtained at 5 kN. This value decreased further at higher loads. The hydraulic grip pressure used for gripping test specimens was 500 psi.

Fig. 2. Schematic drawing of the high temperature mechanical testing unit employed.

To improve thermal stability of the contact extensometer a constant temperature circulator with the capability of maintaining cooling water temperature at ±0.1°C, was used. Moreover, to help mechanical stability of the contact rods on the specimen surface, particularly under cyclic fatigue loads, a spring capable of exerting 600 g force on a specimen through the extensometer rods (300 g for each rod) was installed. As a result, no slippage of the extensometer rods was observed, and thermal stability of the unit was very good. Moreover, the examination of failure locations in the fractured specimens showed that the failure did not originate from the contact areas, indicating that the force level applied by the spring was not detrimental for the current specimen geometry.
All tests were conducted in N₂ atmosphere and under a slightly positive pressure. The specimens were heated to the test temperature of 1300°C in load control using a small load. Prior to tests, the specimens were allowed to equilibrate with the furnace for at least one hour. The monotonic tensile tests were conducted in strain-control with a cross-head speed of 0.1 μm/s, corresponding to a strain rate (ė) of 4x10⁻⁶ s⁻¹. The cyclic tensile tests were conducted in load-control using a sinusoidal wave-form with a frequency of 0.5 Hz and a stress ratio (R=σ_min/σ_max) of 0.1. Microstructural analyses of the test specimens were carried out by optical microscopy, Scanning Electron Microscopy (SEM) and Transmission Electon Microscopy (TEM).

RESULTS AND DISCUSSION

Monotonic Tensile Test

To get experimental parameters for the fatigue tests, first, a monotonic tensile test was conducted. This was done at a low strain rate (4x10⁻⁶ s⁻¹) to allow the composite to redistribute transient stresses as much as possible while loading. Transient stresses originate mainly from the differences in elastic, creep and thermal properties of constituents. Typically, if testing is conducted using a high strain-rate (rapid loading), depending on the magnitude of the differences in above properties, nonuniform stress build-up within the composite could lead to premature failure since there is not enough time for the stress to redistribute itself among different phases.

The uniaxial tensile stress-strain curve obtained at 1300°C is shown in Fig. 3. As marked in the figure, the curve appears to have four distinct regions. It should be mentioned that the features seen in Fig. 3 appear to be typical at elevated temperatures since the curve obtained at 1200°C was similar. First is the elastic region (Region-1), where both the fiber and matrix were strained elastically. The area of Region-1, which represents the amount of elastic energy stored in the specimen, clearly indicates that elastic deformation at 1300°C was very limited. Region-1 ends at the point of deviation from linearity due to the macrocrack formation in the matrix. In composite literature this point is referred to as the proportional limit, σ_pl, and its exact location in the curve depends on the sensitivity of displacement and load transducers used. It was shown by the surface replication [5,6], acoustic emission [6,7] and frictional heating measurement [5] methods that the proportional limit seen in stress/strain curve is not the point where the first matrix crack occurs (microcracking threshold).

Beyond σ_pl (Region-2) the composite became increasingly compliant with stress. In contrast to room temperature tests where the increase in compliance is entirely due to crack formation, the increase at 1300°C is due to both the crack formation and creep since Region-2 in Fig. 3 covers a larger strain range under similar stress levels than at room temperature [8]. At about 160 MPa the decrease in tangential modulus of the composite ceased and is designated as the matrix crack saturation limit, σ_mcs1. At this point matrix cracks are assumed to have fully formed, and further increase in strain was due to the creep of the bridged fibers only (Region-3). The constancy of the slope in Region-3 suggests that the creep rate of the fibers was constant (a steady state regime). Up to this point, the stress/strain curve in Fig. 3 is in
agreement with the prediction by Aveston, et al. [9], where they foresaw the formation of such distinct regions in the curve of unidirectional composites exhibiting multiple matrix fracture.

![Stress/strain curve](image)

**Fig. 3.** Monotonic stress/strain curve of the SiC/SiC composite obtained at 1300°C. Note that the specimen exhibited distinct deformation regions.

At the end of Region-3, the composite became compliant again with increasing stress. The start of this new region (Region-4) is designated as the fiber yielding point, \( \sigma_{fy} \), due to the dominance of creep under increasing stresses. The fiber failure and the pull-out are also expected to account some of the increase in compliance in Region-4, however the relatively large size of Region-4, extending from 1 to 1.5% strain, suggests that the primary event was the creep of the bridging fibers. In the absence of creep, Region-4 probably would not have appeared in Fig. 3 or would have been small since brittle fiber fracture leads to catastrophic composite failure. This is believed to be the reason why the curves obtained at room temperature tests did not have Region-4 [8].

Based on the stress/strain behavior of composite in Fig. 3, the discussion above implicitly indicated that the fibers displayed all three main creep states, i.e, primary (Region-2), steady-state (Region-3) and tertiary (Region-4) stages. This appears to be in contrast to the observations made Bodet, et al. [10] during creep testing of individual fibers between 1200 and 1400°C. They reported occurrence of only primary creep Nicalon fibers in Ar and both the primary and steady-state creep in Ar/CO environment. In some instances, however, they also observed accelerated creep (tertiary) due to necking in Ar/CO environment. The differences indicated between the two studies may be attributed to the fact that the present test was conducted under increasing load and that the test specimen was composite. They carried out tests under constant load and the specimen was fiber. As will be
shown later we have observed an evidence (necking) for the presence of tertiary creep in the composite in Fig. 3.

The stress/strain curve in Fig. 3 in general is similar to the one observed in the SiC/CAS composites although the tests with the SiC/CAS composites were conducted at room temperature [11]. Fig. 3, however, exhibits some important differences with respect to that obtained from the same composite at room temperature [8]. While both 1300°C and room-temperature curves showed almost identical elastic regions (Region-1), inelastic regions and their boundaries were different. For instance, the composite failure at room temperature took place in Region-3 where bridged fibers were deforming elastically. Moreover, the failure strain at room-temperature test was only 1/3 of the strain seen in Fig. 3, yet the overall fracture stress of the composite was almost the same. The fracture stress results (both at 1200 and 1300°C) suggest that the Nicalon fibers which are the primary constituent carrying the load, did not deteriorate at 1300°C in N₂ environment. TEM observations showed that the interfacial carbon was intact and no microstructural change took place in the fibers. Thus, in nonoxidizing environments the short term strength up to 1300°C appears to be independent temperature. In oxidizing atmosphere, however, the fibers were shown to have lost about 45% of their room temperature strength at 1300°C [12].

Fractography showed that the fracture surfaces obtained during monotonic tests were nonplanar and rough, as compared to those obtained during similar tests at room temperature. The fracture surfaces included steps which were parallel to 0°-fibers, indicating presence of shear failure. In microscopic level, the fracture surface also had distinctive features. Fig. 4 shows a typical SEM micrograph obtained from the specimen in Fig. 3. The fibers had debris, both matrix particles and interfacial carbon. Most importantly, they exhibited features which suggest occurrence of necking at this temperature. Thus, the fiber fracture surfaces in general were nonplanar. In spite of evidence that creep took place, TEM studies showed that no microstructural changes occurred in the fibers during the slow strain-rate test in Fig. 3. Since SiC fibers also contain Si-C-O phase, it was suggested in the creep fiber creep studies [10], however, that the viscous flow of this phase is responsible for the long term creep of the Nicalon fibers.

Since the fibers have higher strain to failure, the matrix cracks formed early and the fibers ended up carrying most of the load. During microscopic evaluations by SEM and TEM, it was seen that the fiber/matrix interface often had cracks. These cracks were observed at both the fiber/carbon interface and within the carbon layer. A TEM micrograph showing the crack in the latter case is seen in Fig. 5. The cracks in the specimen tested at room temperature were at the fiber/carbon interface cracks. Thus, it appears that the crack formation within the interfacial carbon is exclusive at elevated temperatures. The cracks similar to that in Fig. 5 sometimes deviated from interface and entered into matrix. These observations explain why the fibers generally had carbon and matrix debris on them. Note that the interfacial cracks mentioned above could not be introduced during the specimen preparation since as-received (untested) specimens prepared in the same way did not exhibit similar fiber/matrix detachment unless the specimen was oxidized, thus the carbon layer was lost.
Fig. 4. SEM micrograph of the fracture surface of the monotonically loaded specimen in Fig. 3. The fibers generally had carbon/matrix debris on them. The distinctive rough fracture surfaces in fibers indicate the occurrence of nonbrittle fracture.

Fig. 5. TEM micrograph from the monotonically loaded specimen in Fig. 3. The crack formation within the carbon layer at the fiber/matrix interface was commonly observed in this specimen.
Fatigue Testing

It was shown experimentally in previous occasions that there is a very good correlation between the increase in crack density and the decrease in stiffness of the composites [13,14]. Thus, in this study the crack evolution under cyclic loads was monitored by the in-situ stiffness measurements.

The maximum stress ($\sigma_{\text{max}}$) values for the fatigue tests were selected based on the monotonic stress/strain curve in Fig. 3. As a result, $\sigma_{\text{max}}$ values of 108, 145, 188 MPa, which correspond to about 50%, 65% and 85% of the Failure Stress (FS) observed in the monotonic test were used. In all three cases the fatigue tests were started from the zero load and continued until the end of test without any interruption. The data acquisition was made in a logarithmic interval. Moreover, to be able to catch the events taking place in the specimen prior to a possible specimen failure, a buffer was used to record the last several cycles. At the end of the tests, only the specimen fatigued with $\sigma_{\text{max}}$=50% FS survived the intended number of cycles of 72 000 (40 hrs). The specimens fatigued with $\sigma_{\text{max}}$=65% and 85% FS failed in 7100 and 890 cycles, respectively. The specimens during these tests exhibited similar behavior. As an example, the stress/strain data obtained during the fatigue test with $\sigma_{\text{max}}$=65% FS is shown in Fig. 6. The individual loading/unloading cyclic curves were recorded throughout the test, but for clarity only the selected curves are included in the figure. Notice that since the test was conducted under constant $\Delta \sigma$, the loading/unloading curves moved to right along the strain axis with increasing fatigue cycles. This is a clear indication that the test specimen accumulated irreversible (residual) strain under cyclic loads at 1300°C. Moreover, the specimen exhibited a hysteresis loop, but its

![Fig. 6. Loading/unloading curves obtained from the specimen fatigued with $\sigma_{\text{max}}$=65% FS (145 MPa). Notice that the specimen crept under the cyclic load.](image-url)
area decreased with fatigue. These two observations at 1300°C are in contrast to those made on the similar composites at room temperature [8,15], where the specimens showed almost no residual strain and hysteresis loops in spite of the presence of matrix cracks.

Fig. 7a shows all the strain data recorded during the fatigue test in Fig. 6. The variations of total, residual and elastic strains as a function of fatigue cycle are clearly seen. The total strain ($\varepsilon_t$) was measured at maximum stress while the residual strain ($\varepsilon_r$) was obtained at zero stress (specimen unloaded). The elastic strain ($\varepsilon_e$) was determined from the relationship in equation (1),

$$\varepsilon_e = \varepsilon_t - \varepsilon_r.$$  (1)

Note in Fig. 7a that due to the decrease in stiffness at the beginning of tests in Fig. 6, the elastic strain increased in small amount but remained relatively constant afterwards. The residual strain, on the other hand, increased rapidly at the beginning to about 1000 cycles and then slowed down with the increasing fatigue cycles (creep curve). As a result, the total strain ($\varepsilon_e + \varepsilon_r$) reflected the change of residual strain during the test. The deformation curve in Fig. 7a is redrawn in Fig. 7b in terms of strain-rate. Initially, the strain-rate was high, between $10^{-4}$ and $10^{-5}$ s$^{-1}$, but dropped quickly as the test proceeded. The decrease in strain-rate was continuous, and a stated-state regime was not reached. This means the region up to the 6000 cycles was still the primary creep area. Beyond 6000 cycles, the strain-rate showed fluctuations, which are believed to be the indications of fiber failure since the specimen failure occurred afterwards. Just before composite fracture the strain-rate exhibited a sudden increase, as expected. From the observations in Fig. 7b it seems that the strain-rate plot is sensitive for the event in the specimen, particularly those taking place before the fracture.

To compare the initial level of damage introduced into the specimens under different loads the first two loading/unloading cycles of three tests are compared in Fig. 8. The origins of the tests were displaced for clarity. The proportional limits in Fig. 8 appear to be the same as the one observed in the monotonic test (Fig. 3). The comparison of slopes of the first and second loading cycles in each test clearly suggests that a significant amount of overall damage was introduced during the initial loading. Moreover, the matrix damage introduced into the specimens increased with the increasing applied load. An important observation made in this study is that for given stress values the fatigue tests were seen to lead to much smaller strain levels in the test specimens than the monotonic tests, i.e. less damage is introduced during the fatigue test. This observation, which can be seen clearly from the comparisons in Figs. 3 and 8, suggests the presence of a strain-rate effect in the SiC/SiC composite since the fatigue tests were typically conducted in strain-rates about three orders of magnitude higher than the monotonic tests.

Fig. 9 shows the normalized stiffness as a function of fatigue cycle in all three tests. Normalized stiffness values were obtained by dividing the instantaneous values by initial stiffness (elastic modulus). The stiffness measurements were made automatically during the tests from the loading portions of the loading/unloading curves. The scatter in stiffness values is believed to be due to the varying degrees
Fig. 7. Change of a) the total strain, residual strain and elastic strain, and b) the strain rate, as a function of fatigue cycle during the test in Fig. 6.
Fig. 8. Comparison of the first two loading/unloading curves obtained in three different specimens where the maximum fatigue stress, $\sigma_{\text{max}}$, was 50% (108 MPa), 65% (145 MPa) and 85% (188 MPa) FS.

Fig. 9. Variation of normalized stiffness as a function of fatigue cycle in all three fatigue tests.

of wedging and bridging in each cycle in this relatively porous composite and, as expected, it decreased with increasing applied stress. The stiffness curves in Fig. 9 show that the damage accumulation occurred in two distinct steps. Up to 100 cycles, the stiffness decreased rapidly in all three cases, but the rate with which this occurred was strongly dependent on the applied stress, increasing with increasing stress. The high-rate region up to 100 cycles is believed to
correspond to crack formations within the matrix of 90°-bundles since the fibers in these bundles are loaded in radial direction and thus are not effective carrying the load. Because the matrix has a very low strain-to-failure, the lateral cracks took place readily and appeared to be reaching a saturation. Furthermore, the strong dependency of stiffness loss on applied stress suggests that the higher loads lead to higher crack density, i.e. reduced crack spacing.

After 100 cycles, however, the decrease in stiffness shown in Fig. 9 leveled off and its rate appeared to be weakly dependent on the applied stress. In this region, the cracks are postulated to be forming within both the 90°- and 0°-bundles. Notice also that the overall stiffness loss at failure does not appear to have any significance for the occurrence of specimen failure, i.e. there is not a specific stiffness loss value at which the failure is expected to take place. For instance, Fig. 9 shows that the specimen fatigued with \( \sigma_{\text{max}} = 65\%\) FS failed after it had about 60% of the initial stiffness at 7100 cycles, while the specimen with \( \sigma_{\text{max}} = 85\%\) FS failed after it had about 30% of the initial stiffness at 890 cycles. The test with \( \sigma_{\text{max}} = 50\%\) FS survived the 72000 cycles. It is clear that both the normalized stiffness and the fatigue life decreased with increasing applied stress level. This implies that both the matrix cracking and the fiber damage increased with the fatigue stress since the matrix is primarily responsible for the stiffness loss and the fiber damage is the one which determines the fatigue life, at least in inert atmospheres.

The optical micrograph in Fig. 10 shows the typical crack pattern developed in the composite subjected to fatigue with \( \sigma_{\text{max}} = 85\%\) FS. The 90°-bundles in this figure are perpendicular to the micrograph; thus the load was applied along the longitudinal fibers in Fig. 10. As a result, the lateral matrix cracks occurred primarily within the 90°-laminates. Except for the crossing points at the 0°-fiber bundles the cracks were relatively continuous across the specimen thickness. At crossing points the matrix cracks were deflected and thus, the 0°-fibers were generally intact. The average crack spacing in this specimen was about 300 \( \mu \text{m} \), corresponding to a crack density of 3.3 \( \text{mm}^{-1} \).

In general, the fracture surfaces of the fatigued specimens were smoother than those obtained under monotonic loading. Moreover, at microscopic level the brittle fracture features were observed. The SEM image in Fig. 11 is from the specimen fatigued with \( \sigma_{\text{max}} = 85\%\) FS. The features seen in this micrograph are typical for the fatigued specimens: the fiber surfaces in general are smooth and the fracture surfaces of the fibers are relatively flat, indicating the dominance of brittle fracture rather than creep failure. Both of these observations are in contrast to those made on the fracture surface of the monotonically fractured specimen, where rougher specimen and the fiber fracture surfaces were seen. Since both the monotonic and fatigue tests were conducted at the same temperature and creep took place in both cases, the differences in the observations are believed to be due to the differences in the rate of loading in these two tests. Since the fatigue tests were conducted in a faster rate and the brittle fracture seemed to be the fracture mode, these observations in the SiC/SiC composite at 1300°C are similar to those in metals. In metallic materials, it is well established that high strain-rate promotes brittle fracture because the dislocations are relatively immobile in these tests.
Fig. 10 Lateral crack pattern obtained in the specimen fatigued with $\sigma_{\text{max}}=85\%$ (188 MPa) FS. The cracks developed mostly within the $90^\circ$-fiber bundles and had about 300 $\mu$m spacing.

Fig. 11 SEM micrograph of the fracture surface of the fatigued specimen with $\sigma_{\text{max}}=85\%$ (188 MPa) FS. The fiber surface became smooth due to the repetitive loading. The commonly seen flat fiber fracture surface suggest the dominance of brittle fracture.
CONCLUSIONS

1. The monotonic stress/strain curve of the SiC/SiC at 1300°C exhibits distinct regions. Matrix cracks first develop within the 90°-bundles and as the crack density saturates the load is carried primarily by the bridging fibers. Eventual composite failure occurs by the rupture of fibers. While the composite fracture stress at 1300°C is similar to that at room temperature, the fracture strain increases by about 200%, clearly illustrating the effect of creep at this temperature.

2. The creep is also prominent under cyclic loads. The overall crack density increases and the fatigue life decreases with the applied cyclic stress. Up to 100 cycles stiffness loss strongly depends on the applied stress; beyond that, however, the dependence is weak.

3. The fracture surfaces of the monotonically loaded and fatigued specimens are different. Although creep takes place in all specimens, because of the difference in strain-rates the final fracture under monotonic load occurs by the creep of fibers while that under cyclic load takes place by brittle fracture of the fibers.

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