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INTERFACE OXIDATION AND STRESS-RUPTURE OF Nicalon™/SiC CFCCs AT INTERMEDIATE TEMPERATURES

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ABSTRACT

The effect of oxidation of the carbonaceous fiber coating on the intermediate temperature stress-rupture behavior of a Nicalon™/C/SiC continuous fiber composite was modeled. The model, that was reduced to the analysis of a general ideal bundle composed of classical fibers subjected to constant loading, predicts that the oxidation of the fiber coating triggers a sequence of processes that can lead, under certain conditions, to composite failure. These processes involve loss of stress transfer between the fiber and the matrix, fiber overloading, and fiber failure. The implications of the model predictions are discussed in relation to experimental measurements at 425°C in air that show that Nicalon™/C/SiC exhibits time-dependent loss of strength.

1. INTRODUCTION

Reliability and durability are two of the main considerations in the selection of materials for long-service life components and applications. For example, for those applications considered in the U. S. Department of Energy-sponsored Continuous Fiber Ceramic Composite (CFCC) Program [1], where components are expected to perform continuously for tens of thousands of hours while subjected to relatively constant stress levels, the durability of candidate materials has been typically investigated through stress-rupture testing. Stress-rupture test strategies are usually designed around tests at the highest expected service temperatures, assuming, sometimes erroneously, that the severity of the experimental conditions is proportional to the test temperature. Recent results from the evaluation of Nicalon™/SiC composites in air at temperatures as low as 425°C have validated this observation, and have redrawn attention at the same time, to an old but potentially serious problem stemming from the poor performance of these materials at so-called intermediate temperatures [2,3]. Although, during service, CFCC components are envisioned to be exposed to the highest temperatures in the system, typically between 900°C and 1200°C for most applications, the need to join CFCC parts to other system components implies that often portions of the CFCC will be exposed to intermediate temperatures. Similarly, exposure to intermediate temperatures will occur during transients and during system shut downs and start ups.

The first indications of this intermediate-temperature problem, sometimes referred to as "intermediate temperature embrittlement", were documented when the flexural strength of Nicalon™/glass-ceramic CFCCs, evaluated between room temperature and 1200°C, was found to reach minimum values between 600°C and 700°C [4]. More recent studies have shown that even static oxidation treatments at temperatures as low as 300°C can lead to the embrittlement of Nicalon™/CAS [5]. Stress-rupture tests conducted in flexure at stresses larger than the matrix cracking stress and at temperatures between 300°C and 1100°C, have shown that Nicalon™/SiC CFCCs with carbonaceous fiber coatings exhibit time-dependent loss of strength [2]. Similar results have been reported for tests conducted in tension [3].

Models of the creep-rupture of ceramic composites have been formulated to address the cases of crack growth in an elastic matrix reinforced with fibers that creep linearly with stress [6], or that of a composite reinforced with fibers that break down with time and

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stress [7]. In both cases, these models assume that load transfer between the fibers and matrix is constant and time independent. Based on the interpolation and extrapolation of experimental data bases using the "micro-kinetic approach", models have also been developed to predict the remaining strength and life of ceramic composites in the presence of generalized time-dependent degradation processes [8]. However, none of these models considered the effect of fiber coating oxidation on the stress-rupture behavior of CFCCs.

This paper begins by summarizing experimental stress-rupture results obtained at 425°C in air for a Nicalon™/C/SiC CFCC. Then, a model is formulated to calculate the probability of failure of such composites when oxidation of the carbonaceous fiber coating occurs. As will be shown, this formulation was reduced to the analysis of a general ideal bundle composed of classical fibers, with the features of Daniels' classic fiber model [9]. The implications of the model predictions are identified in relation to the experimental results. The limitations of the model are also addressed and additional considerations are discussed.

2. EXPERIMENTAL PROCEDURES

Dog bone-shaped tensile specimens, 200 mm long, 3.5 mm thick, and 10.2 mm wide in the gauge section were used in the experimental part of this investigation. The tests were conducted using a universal testing machine equipped with a compact furnace that produced a uniformly heated zone of 30 mm along the gauge length of the specimen. Aluminum end-tabs were adhesively bonded to the specimens, which were gripped using water-cooled hydraulically actuated grips. The specimens consisted of plain-weave ceramic-grade Nicalon™ fiber fabric, coated with a 0.3 μm thick layer of carbon. Both the carbonaceous fiber coating and the SiC matrix were densified by chemical vapor infiltration and the samples were seal-coated using a glassy coating of proprietary composition[†]. The volume concentration of fibers and porosity were 45% and 20%, respectively. A constant load of 50 N was maintained in the load train while the test specimens were heated at a uniform rate of 20°C/minute to the test temperature. After a 20-minutes soaking period, the specimens were mechanically loaded at a constant rate of 10 N/sec to the test load and then the load was kept constant for the duration of the test. The longitudinal deformation of the specimen was monitored continuously during the test using a low-contact force capacitance extensometer over a gauge length of 25 mm.

3. EXPERIMENTAL RESULTS

Figure 1 shows the strain histories recorded during stress-rupture tests conducted in air at 425°C and stresses of 100, 150 and 200 MPa. The strain vs. time curves in Figure 1 are similar in shape and exhibit three well defined stages. Stage I is very brief and coincides with the mechanical loading of the specimen. Stage II is characterized by a regime of decreasing rate of deformation, while, in Stage III, the rate of deformation increases, preceding the failure of the specimen. The lives of the specimens tested at 100 MPa, 150 MPa and 200 MPa were 511.5 hrs, 209.1 hrs and 152.4 hrs, respectively. All three specimens failed in the uniformly heated gauge section and exhibited fiber pull-out lengths in the order of millimeters, in contrast to as-received specimens evaluated at room temperature which showed fiber pullout lengths in the order of hundreds of microns. Figure 2 shows a scanning electron micrograph of the fracture plane of the specimen evaluated at 100 MPa, illustrating the magnitude of fiber pull-out.

In the next section, a simple model is presented as a first attempt to explain the increase of specimen compliance with time, the shape of the strain vs. time curves shown in Figure 1, and the mechanisms leading to the failure of the specimens. The model is based on a sequence of events at the fiber-matrix level that include matrix cracking, the oxidation of the carbonaceous fiber coating, and the statistics of progressive fiber failure.

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4. MODEL

Although the materials evaluated had a 2-D fiber architecture, the model presented here has been simplified by considering an idealized unidirectional composite. The implications of this simplification will be discussed later in the paper. Let us consider, then, that our ideal unidirectional composite is reinforced with fibers of uniform diameter $2r$ that occupy a fraction v_f of the composite volume. It will be assumed that the fibers are coated with a thin compliant layer of carbon that forms low fracture toughness interfaces between the fibers and the fiber coating, and between the fiber coating and the matrix. Let us also assume that the composite is subjected to a constant tensile stress σ_c , that was applied "instantaneously" such as during a stress-rupture test. When the applied stress σ_c exceeds the matrix cracking stress, a series of parallel cracks spaced l_c apart, will form in the matrix. Without losing generality, let us assume that the relationship between the density of transverse cracks in the matrix and the applied composite stress can be described by the diagram in Figure 3. At the plane of the matrix cracks, the axial stress in the fibers will increase to support the entire stress applied to the composite. Moving away from the crack surfaces, the average fiber axial stress decays linearly at a rate of $2\tau/r$ as the matrix picks up the complementary portion of the load, where τ is the average interfacial shear stress. The axial stress distribution along the fiber will then be given by

$$\sigma_z^f(z) = T - \frac{2\tau z}{r} \quad 0 \leq z \leq l_c/2 \quad (1)$$

where T is the axial stress in the fibers at the crack plane. Figure 4 shows a diagram of the axial stress profile in the fibers. If we assume global load sharing conditions, then the relationship between the maximum stress in the fiber T , and the constant applied stress to the composite σ_c will be given by:

$$\sigma_c = v_f T (1 - \Phi) \quad (2)$$

where Φ is the fraction of failed fibers, i. e., $\Phi = 1 - (N/N_0)$, N being the number of surviving fibers and N_0 the original number of fibers. As a consequence of the global load sharing assumption, the maximum stress in the surviving fibers, T , will increase with progressive fiber failure as dictated by Equation 2, even though the value of σ_c remains constant.

If we assume that the strength of the fibers is described by a two parameter Weibull distribution, and that the strength-controlling flaws are restricted to the surface of the fibers, then the probability of failure of the fibers will be given by [10]:

$$\Phi = 1 - \exp \left\{ - \int_{\text{area}} \frac{1}{A_0} \left(\frac{\sigma(z)}{\sigma_0} \right)^m dA \right\} \quad (3)$$

where σ_0 is the Weibull characteristic strength for a fiber of surface area $A_0 = 2\pi r l_0$ loaded in uniform tension, and m is the Weibull modulus. In this case, σ_0 is the stress required to cause on average, one failure in a fiber of length l_0 [11].

Considering the periodicity in the matrix cracks spacing, the probability of failure of a fiber over the entire gauge length (h) of the tensile specimen will be given by:

$$\Phi = 1 - \exp \left\{ -2 \frac{1}{\sigma_o^m l_o} \frac{h}{l_c} \int_0^{l_c/2} (\sigma(z))^m dz \right\} \quad (4)$$

where h is the specimen's length and h/l_c is the number of matrix blocks in the specimen (see Figure 4). The factor of two in Equation 4 is introduced because the integration is carried out up to $l_c/2$ to take advantage of the symmetry of the fiber axial stress profile with respect to the matrix crack plane. After carrying out the integration in Equation 4 using the expression for the fiber's axial stress profile (Equation 1), we obtain:

$$\Phi = 1 - \exp \left\{ -\frac{h}{l_c} \left(\frac{T}{T_o} \right)^{m+1} \left(1 - \left(1 - \frac{\tau l_c}{r T} \right)^{m+1} \right) \right\} \quad (5)$$

$$\text{where } T_o = \left(\frac{(m+1) \sigma_o^m \tau l_o}{r} \right)^{\frac{1}{m+1}} \quad (6.a)$$

and

$$\frac{\tau l_c}{r T} < 1 \quad (6.b)$$

Because Equations 2 and 5 are not independent, it follows that for a given constant stress σ_o , the specimen will either achieve an equilibrium state (if there is at least one value of T that satisfies both Equations 2 and 5) or it will fail by rapid progressive fiber failure in a process analogous to an "instantaneous" domino effect¹, in which case, there isn't a value of T that simultaneously satisfies Equations 2 and 5. These conditions will be illustrated in the next section.

Let us assume that our idealized unidirectional composite is heated in air at a temperature where the carbonaceous fiber coating oxidizes but without the formation of oxide species on the fiber or matrix. For example, for a NicalonTM/C/SiC composite, 425°C may be such a temperature. Furthermore, at this temperature, none of the composite constituents is expected to exhibit time-dependent deformation (meaning creep or stress relaxation). If we assume that due to oxidation, the carbonaceous fiber coating recedes along the length of the fibers at a rate of

$$\frac{d\zeta}{dt} \quad (7)$$

where ζ is the recession length of the carbonaceous fiber coating, then the stress profile along the fibers (Equation 1) will become time-dependent and will be given by:

$$\sigma_z^f(z, t) = T(t) [U(z)] - \frac{2\tau}{r} (z - \zeta(t)) [U(z - \zeta(t))] \quad 0 \leq z \leq l_c/2 \quad (8)$$

¹ In fact, it is more like a "snow ball" effect because the stress on the surviving fibers continuously increases as other fibers fail, until all fibers fail.

where $[U(z - z_0)]$ is Heaviside's unit function defined as

$$[U(z - z_0)] = \begin{cases} 1 & z > z_0 \\ 0 & z < z_0 \end{cases} \quad (9)$$

After substituting Equation 8 into Equation 5 and carrying out the integration we obtain:

$$\Phi = 1 - \exp \left\{ -\frac{2h}{l_c} \left(\left(\frac{T(t)}{\sigma_0} \right)^m \frac{\zeta(t)}{l_0} + \frac{1}{2} \left(\frac{T(t)}{T_0} \right)^{m+1} \left(1 - \left(1 - \frac{\tau}{T(t)r} (l_c - 2\zeta(t)) \right)^{m+1} \right) \right) \right\} \quad (10)$$

which is the fraction of failed fibers as a function of time. The average axial strain of the composite will be given by the strain in the fibers, which can be determined by integrating the fiber axial stress along the length as follows:

$$\epsilon(t) = \frac{2}{E_f l_c} \int_0^{l_c/2} \sigma_z^f(z, t) dz \quad (11)$$

Because of the loss of the fiber coating due to oxidation, the mechanism of stress transfer between the fiber and the matrix will be inhibited. This will result in subjecting larger portions of the fibers to the peak stress, T (see Figure 5) and consequently in increasing their probability of failure (Equation 10). The occurrence of these processes will also result in an increase in the specimen compliance.

To generate some model predictions, it is necessary to know the relationship between the applied stress and the matrix crack density, the fiber strength distribution, the interfacial shear stress, and the rate of oxidation of the carbonaceous fiber coating. Following the analysis of Filipuzzi and Naslain [12], the following considerations were made to calculate the rate of recession of the carbonaceous fiber coating due to oxidation. Assuming that the oxidation of carbon in air occurs according to²

then, at temperatures below 700°C, the oxidation of the carbonaceous fiber coating is controlled by interface reaction with the intrinsic reaction constant given by [12]

$$V_i \text{ (kg} \cdot \text{m}^{-2} \cdot \text{s}^{-1}) = 1070 \exp \left(-\frac{123,000}{R\theta} \right) \quad (13)$$

where θ is the absolute temperature. At 425°C, it is found that the rate of carbon recession, $d\zeta/dt$ is given by

$$\frac{d\zeta}{dt} = \frac{V_i}{\rho_c} = 3.5 \times 10^{-10} \text{ m/sec} \quad (14)$$

² Whether the reaction is $C + O_2 \rightarrow CO_2$ or $2C + O_2 \rightarrow 2CO$, is not an important consideration here.

where ρ_c is the density of carbon ($\approx 2.2 \text{ g/cm}^3$). The magnitude of the rate of carbon recession given by Equation 14 is consistent with analyses and data reported elsewhere [for example see ref. 13]. It is important to point out that the rate of carbon recession is independent of geometry or the thickness of the fiber coating. The values of other parameters used in the calculations have been summarized in Table 1.

5. MODEL PREDICTIONS

Figure 6 illustrates the algorithm used in the calculations. Although in principle this algorithm is quite simple, it is described here because it reveals some interesting characteristics that are intrinsic to the mechanics of the problem. Both Equations 2 and 5 have been plotted in Figure 6 for $\sigma_c = 195 \text{ MPa}$ and $\sigma_c = 225 \text{ MPa}$. The algorithm starts with the calculation of T based on Equation 2, assuming that $\Phi = 0$ (no failed fibers). In Figure 6, these points correspond to $T = 780 \text{ MPa}$ and $T = 900 \text{ MPa}$ respectively. With this value of T , Φ is determined using Equation 5, and a new value of T is calculated from Equation 2 using the value of Φ just found. The process is repeated until either the iterated function system settles at the attractor, which coincides with the smallest value of T that satisfies both Equations 2 and 5, or the iterated system diverges, indicating that there is no value of T that satisfies simultaneously Equations 2 and 5. In the first case, the composite (surviving fibers) can handle the load without further fiber failure, whereas in the latter, a domino effect is generated resulting in the rapid failure of all the fibers. In both cases, this initial process occurs "instantaneously" upon the application of the load. Using this algorithm, the strain histories of the composite were predicted for different values of σ_c , but this time using Equations 2 and 10 as shown in Figure 7³. The predicted composite strain in Figure 7 was normalized by $\sigma_c/v_f E_f$ which would be the strain in the composite if all the fibers were intact and were carrying all of the load. The time has also been normalized in Figure 7 so that at normalized time equal to one, all the carbon has been consumed. The normalized strain at time zero is not equal to one because the model accounts for both the reduced strain in the fibers along the stress-transfer zone and the failure of fibers upon the "instantaneous" application of the composite stress σ_c .

The results in Figure 7 indicate that the composite can either achieve an equilibrium state after all the carbon has been consumed and the deformation stops with no more fibers failing ($\sigma_c < 195 \text{ MPa}$) or it becomes unstable during the oxidation of the carbonaceous fiber coating, resulting in composite failure ($\sigma_c \geq 195 \text{ MPa}$). The process leading to this instability during the oxidation of the carbon coating is illustrated in Figure 8 for the case when $\sigma_c = 200 \text{ MPa}$. In this case, it can be observed that initially the composite (fibers) can withstand the instantaneous application of the load, as demonstrated by the fact that the curves associated with Equations 2 and Equation 10 intersect when $\zeta = 0$. As time progresses, and ζ and T increase, the number of failed fibers also increases. This is represented by the translation of the curves associated with Equation 10 in the Φ - T plane. Furthermore, with the translation of these curves, the position of the attractor will also change. Eventually, the curves associated with Equations 2 and 10 will not intersect anymore at which point, the composite fails. This is demonstrated for example when all the carbon has been consumed ($\zeta = 1/2$). It is interesting to observe that for all the conditions considered in these examples, it only takes the failure of a relatively small fraction of the fibers to trigger composite failure.

6. DISCUSSION

The sequence of mechanisms assumed in the model appear to be consistent with the microstructural features observed in the fracture surfaces of the specimens (Figure 2), particularly the extremely long fiber pull-out lengths resulting from the removal of the carbonaceous fiber coating. This observation is also in agreement with the formation of

³ Equation 5 and 10 are equivalent when $t = 0$ and $\zeta = 0$.

channels, between fibers and matrix, that has been reported from the microstructural analysis of similar materials subjected to thermomechanical treatments at the same temperature regime [14].

Figure 9 shows a comparison of the predicted and experimentally recorded strain histories. It can be observed that the predicted and recorded strains are of the same order of magnitude, and that although the time scale predicted by the model for complete carbon burnout is also comparable to the experimental failure times, the model doesn't predict failures for stresses below 195 MPa, while such are experimentally observed. There are several possible reasons for this discrepancy. First of all, by considering a unidirectional composite with equally distributed fibers of equal diameters, the model assumes a very idealized situation. In actuality, because the fibers are part of a complex weave architecture they are not straight, and therefore are not subjected to uniform stresses. A more realistic model should consider therefore, the effect of fiber bending, particularly at the points of tows cross-overs in the weave, on the fiber stresses. However, although the addition of this consideration will result in the prediction of delayed failures at stresses below 195 MPa, it will also result in the prediction of lower "instantaneous" strengths.

Evidently, the model predictions depend on the value of the parameters used in the calculations, particularly on those parameters associated with the strength distribution of the fibers. For example, by decreasing the Weibull modulus or the characteristic strength, both instantaneous and delayed failures are predicted at lower composite stresses. Similarly, the failure times can be modified by changing the relationship between the matrix crack spacing and the composite stress. However, when using realistic values for all the model parameters, while trying to maintain constant certain relationships, such as the one between crack spacing and interfacial shear stress for example, it was not possible to simultaneously predict delayed-failures for stresses between 100 and 195 MPa within the experimentally observed time scale. The most likely explanation for this inability of the model to predict the experimental results is that the range of stresses for which the oxidation of the fiber coating can trigger composite failure is very narrow. At stresses below the lower limit of this range the composite never fails, whereas for stresses higher than the upper limit of the range, the composite fails upon the "instantaneous" application of the load. For the results shown in Figure 7, the range of stresses for which delayed failures are predicted by the model is only between 195 MPa and 220 MPa. Therefore, it appears that, in addition to the mechanisms considered in this model, it is necessary to consider mechanisms that would be responsible for time-dependent loss of strength of the fibers. Although the suspected mechanisms responsible for such time-dependent fiber strength loss are unknown, it is likely that they will be operational once the fiber coating is removed.

Studies of the effect of oxidation on the mechanical properties of Nicalon™ fibers suggest that microstructural changes occur when the fibers are exposed to air at intermediate temperatures [15-16]. It is suspected that these changes, which result from the decomposition of the fiber due to its unstable nature, are most damaging at intermediate temperatures when the fibers are unable to form an oxide layer that would slow the rate of fiber outgassing. Since the effect of these mechanisms on the degradation of strength is likely to be enhanced under stress, the model needs to be reformulated by incorporating fatigue effects as described originally by Coleman in his classic work on the statistical breakdown of fibers and bundles [17]. By adding this feature, it will be possible to expand the range of stresses for which the model will predict composite failures, while by adjusting the value of the rate of fiber strength loss, it will be possible to predict failure times within the experimental time scale. The formulation of such a model and the microstructural characterization of Nicalon™ fibers subjected to stress-rupture testing at intermediate temperatures are the subjects of on-going research.

7. CONCLUSIONS

Based on a sequence of events that include matrix cracking, oxidation of the fiber coating, fiber overloading and fiber failure, a model was developed to account for the time-dependent changes in compliance that are observed when Nicalon™/C/SiC CFCCs are loaded in air at 425°C under a constant tensile stress. The model predicts that the burnout of the carbonaceous fiber coating triggers a sequence of mechanisms that result in fiber overloading, and under certain conditions, the delayed failure of the composite. The time-scale predicted for the complete removal of the carbonaceous fiber coating and the predicted composite elongation are comparable with experimental values of the time to failure and composite strain, respectively. However, when the composite is evaluated under stress-rupture conditions, the range of stresses over which the model predicts delayed-failure is much narrower than what is observed experimentally. Additional considerations for the occurrence of failures at lower stresses, perhaps by including fatigue effects on the fibers need to be examined.

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r	8.0 μm
τ	4 MPa
v_f	0.25
h	100 mm
m	3.5
l_o	25 mm
σ_o	1800 MPa
h	50 mm
E_f	200 GPa

Table 1. Numerical values used in the calculations.

Figure 1. Strain vs. time record of stress-rupture tests in air at 425°C for a Nicalon™/C/SiC 2-D CFCC.

Figure 2. Scanning electron micrograph of the fracture surface of a Nicalon™/C/SiC CFCC tested at 425°C in air and 100 MPa.

Figure 3. Hypothetical matrix crack density vs. stress relationship for a Nicalon™/SiC.

Figure 4. Schematic of crack distribution in a unidirectional CFCC. The axial stress profile in the fiber is also shown.

Figure 5. Schematic of changes in the axial stress profile in the fiber due to the oxidation of the fiber coating.

Figure 6. Description of the algorithm used in the calculations. The plot is of the probability of failure (or fraction of failed fibers) vs. maximum tensile stress in the fibers at the plane of the matrix cracks.

Figure 7. Predicted strain histories for model composite due to the oxidation of the carbon coating.

Figure 8. Translation of the probability of failure curves with time as a result of the oxidation of the carbon coating.

Figure 9. Comparison of the experimental and predicted strain histories. Although the time scale for complete carbon burnout and the predicted strains are comparable to the failure times and the experimental strains, respectively, the model predicts failures over a very narrow stress range.

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