

# STRESS-RUPTURE, OVERSTRESSING AND A PROPOSED NEW METHODOLOGY TO ASSESS THE DURABILITY AND RELIABILITY OF CERAMIC MATRIX COMPOSITES AT ELEVATED TEMPERATURES

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**SUMMARY:** A new testing strategy is proposed to assess the durability and reliability of non-oxide continuous fiber-reinforced ceramic composites for high temperature structural applications. The strategy is based on determining the reliability (probability of failure) of these materials when subjected to random loading schedules consisting of load and temperature spikes that are superimposed on otherwise constant stress and temperature histories. The frequency and magnitude of the load and temperature spikes would be representative of the number and characteristics of the transients that are associated with a particular industrial application and that are expected to occur over the life of the component. The effect of overstressing on the stress-rupture behavior of a CG-Nicalon™ fiber-reinforced SiC composite was investigated and results are presented from tests conducted in ambient air at 950°C.

**KEYWORDS:** ceramic-matrix composites, durability, reliability, high temperature, stress-rupture, testing

## INTRODUCTION

The development of continuous fiber-reinforced ceramic-matrix composites (CFCCs) has been driven by the promise of substantial economic and environmental benefits if these materials are used in aerospace and energy-related industrial applications [1]. Many of the potential applications for CFCC components (e.g., filters and heat-exchangers in coal-fired power plants, combustor liners in gas turbine engines) are characterized by aggressive environments at elevated temperatures and service lives that are measured in tens of thousands of hours [1].

As CFCCs continue to mature, there is a growing need for long-term thermomechanical property databases and life prediction and design methodologies. Because non-oxide CFCCs exhibit distinct behaviors at stresses above and below the so-called proportional limit stress, there are also questions about the best ways to evaluate these materials and in turn how to use those results for life-prediction purposes. Figure 1 is a typical tensile stress-strain curve for a 2-D CFCC obtained by monotonically loading/unloading the specimen to/from increasingly larger stress levels after each cycle. The proportional limit stress ( $\sigma_{pl}$ ) is associated with the onset of non-linear stress-strain deformation and with the formation of hysteresis loops that result from matrix cracking and frictional sliding of the fibers bridging those matrix cracks. In the absence of environmentally-stable fibers and fiber coatings in oxidizing environments, the proportional limit

stress has long been considered the maximum allowable design stress for non-oxide CFCCs in applications involving oxidizing environments.

Traditionally the long-term durability and reliability of candidate materials for elevated temperature applications, where service stresses and temperatures are more or less constant, has been determined through stress-rupture tests at temperatures and stresses that bracket the expected service conditions. However, experience demonstrates that even in applications with design stresses and temperatures that are constant, components invariably will be subjected to thermal and mechanical excursions outside the design conditions. In the case of non-oxide CFCCs, even if the constant service/design stress is lower than the proportional limit stress, thermal and mechanical excursions could result in overstressing the material beyond the proportional limit stress, even if for short periods of time. Such occurrences would result in the formation of matrix cracks that serve as avenues for the ingress of the environment to the interior of the composite. Depending on the concentration of oxidizing species in the environment, the temperature, the magnitude of overstressing and the ability of the matrix (or lack of) to heal cracks, overstressing could lead to the failure of the composite.

Based on these observations it is clear that for non-oxide CFCCs the application of test methodologies based on traditional stress-rupture testing at stresses larger than the proportional limit stress will yield extremely conservative life data, whereas stress-rupture testing at stresses lower than this stress level will yield overly unrealistic results. To overcome these limitations a new methodology based on random overstressing is proposed to simulate and assess the effects of transients that are characteristic of a specific application and that may subject CFCCs to stresses beyond the proportional limit stress. In the next sections this testing methodology is described and illustrated by evaluating the effect of overstressing on the stress-rupture behavior of a CG-Nicalon™ fiber-reinforced SiC CFCC in ambient air at 950°C.

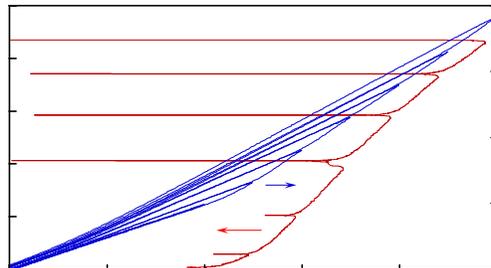


Figure 1. Stress-strain-acoustic emission count plot obtained during the tensile loading/unloading evaluation of a 2-D CFCC. Each loading/unloading cycle was obtained at an increasingly larger stress level. Note that the proportional limit stress coincides with the onset of non-linear behavior, acoustic emission activity and formation of hysteresis loops [2].

## METHODOLOGY

The basic premise of the proposed methodology is to obtain reliability (probability of failure) data for the material under study when subjected to a loading schedule that is representative of the envisioned industrial application. Figure 2 is a schematic representation of such a schedule. The schematic in Figure 2 shows the thermal and mechanical histories of a hypothetical application. Both histories include overstressing conditions which are simulated by the

application of random stress and temperature “spikes”. These spikes are superimposed on otherwise flat profiles of stress and temperature (design stress and temperature). The magnitude and “frequency” of these spikes would be representative of the transients associated with that particular application. For example, in reference to Figure 2, in the case of a component in a gas turbine engine, stress and temperature spikes would be representative of the deviations from the design stress and temperature during start ups (a), shut downs (b), flame outs, foreign object impact (c), etc. The magnitude of the spikes would be estimated from thermal and stress calculations associated with these events, whereas their frequency would be related to the total number of these incidents that occur over the life of the component. This information should be readily available from operating histories of gas turbine engines and other components. Another example is that of filters in coal-combustion or coal-gasification power plants. In this case, the filters also are subjected to transients associated with the operation of the plant in addition to the steady state operating conditions of stress and temperature that include periodic backpulse cleaning which is represented by the cyclic changes (d) in stress and temperature in Figure 2.

Another consideration when using certain non-oxide CFCCs in these applications is the fact that the proportional limit stress may change with time as a result of environmental degradation or from redistribution of internal stresses in the composite [3-4]. This has been represented in Figure 2 as a decreasing proportional limit stress with time.

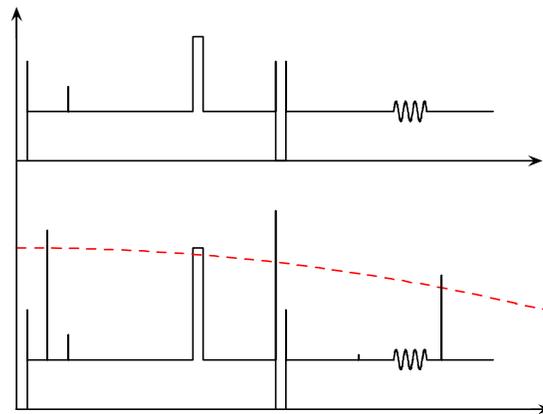


Figure 2. Schematic representation of conceptual thermal and mechanical *random* loading schedules for conducting modified stress-rupture tests. The proportional limit stress  $s_{pl}$  can decrease with time because of environmental effects or redistribution of internal stresses.

## EXPERIMENTAL

The material evaluated consisted of CG-Nicalon™ [0/90] plain weave fabric, coated with a 0.1  $\mu\text{m}$ -thick layer of pyrocarbon prior to matrix densification. The SiC matrix was densified by chemical vapor infiltration (CVI) and contained an enhancement of proprietary composition [5]. The total volume fraction of fibers was 40%. The tensile specimens had a reduced gauge section and an outer seal coating of SiC that was applied by chemical vapor deposition (CVD) after the specimens were machined. The width of the gauge section contained 3 wavelengths of the fabric weave pattern.

Tensile stress-rupture tests were conducted per ASTM C1337 [6] in ambient air (24-40 % relative humidity) using an electromechanical test machine equipped with hydraulically-actuated grips. Specimen deformation was monitored over a uniformly-heated 25 mm-long gauge length using a low-contact force capacitance extensometer. The alignment of the load train was verified prior to each test using a strain gauged metallic test specimen per ASTM 1295 [7]. The grip wedges were water-cooled to preserve the adhesively bonded aluminum end-tabs that were used to minimize specimen damage during gripping. The specimens were heated from ambient temperature up to 950°C using a compact resistance-heated furnace at a constant rate of 20°C per minute. This was followed by a 20 minute soaking period at which point the specimens were loaded at a constant rate of 5 MPa per second up to the test load. In some cases, the load was maintained constant for the duration of the test, whereas in other cases stress spikes were applied at a rate of 10 MPa per second. The tests were completed when the specimens failed.

## RESULTS AND DISCUSSION

Figure 3 shows the strain histories of stress-rupture tests conducted at stress levels of 80, 100 and 120 MPa. In the three cases the applied stress was larger than the proportional limit stress of the material [8]. The resulting strain vs. time curves show that the material became increasingly compliant with time, and that it exhibited accelerated deformation prior to failure. Both the increasing compliance and accelerated deformation of the material prior to failure result from the progressive breakdown of the fiber bundles in the composite as detailed elsewhere [8-10].

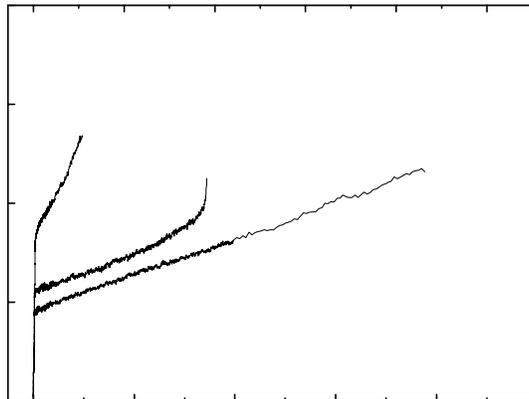


Figure 3. Strain histories of stress-rupture tests of CG-Nicalon™/SiC conducted in ambient air at 950°C at stress levels of 80, 100 and 120 MPa.

The stress-rupture results summarized in Figure 3 were used as a base-line to evaluate the effect of overstressing on the stress-rupture behavior of the material. Evidently, when applying the proposed methodology to a real case it would be necessary to conduct stress-rupture tests at stresses at which specimens would survive for periods of time longer than the envisioned life of the component (i.e., stress-rupture tests at stresses below the proportional limit stress). However in this study a stress level of 80 MPa, which is larger than the proportional limit stress (70 MPa), was used as a baseline stress for illustration purposes. The objective of this exercise was to investigate the effect of stress spikes of 20 MPa and 40 MPa when superimposed on an otherwise constant stress of 80 MPa, by comparing the obtained life times with those determined from regular stress-rupture tests at 80, 100 and 120 MPa.

Figure 4 shows the stress and strain histories of a stress-rupture test at 80 MPa with one spike of 20 MPa applied at the first hour of the test. The stress spike is clearly visible in the stress and strain histories. Also, note that the resulting strain history is similar to those shown in Figure 3 which were obtained from regular stress-rupture tests, and that the application of a single spike of 20 MPa has little effect on the life of the material (22.7 hours vs. 21.6 hours) when not subjected to any spikes. However there is a significant effect on the life of the composite upon the application of a single stress spike of 40 MPa. Figure 5 shows the stress and strain histories associated with a test at a constant stress of 80 MPa and one stress spike of 40 MPa applied after the first hour of the test. In this case the life of the material was reduced from 21.6 hours to 17.6 hours. The effect of overstressing is even more significant when applying multiple stress spikes. Figure 6 shows the stress and strain histories of a test where stress spikes were applied every hour after the first hour of the test for the duration of the test. In this case, the specimen failed shortly after the application of the eighth stress spike and the life of the composite (8.45 hours) was less than one half of the life of the specimen subjected to a constant stress of 80 MPa. However it was three times longer than the life of a specimen subjected to a constant stress of 120 MPa.

The results from these tests are summarized in Figure 7 in a plot of stress versus time-to-failure. In this plot the closed circles represent the results of traditional stress-rupture tests at stresses of 80, 100 and 120 MPa and correspond to the tests summarized in Figure 3. As indicated in Figure 7 the strength of the material under normal stress-rupture conditions decreases with time as

$$\sigma \approx t^{-\frac{1}{4}} \quad (1)$$

which has been explained as the reduction of the fibers strength due to oxidation [8-10].

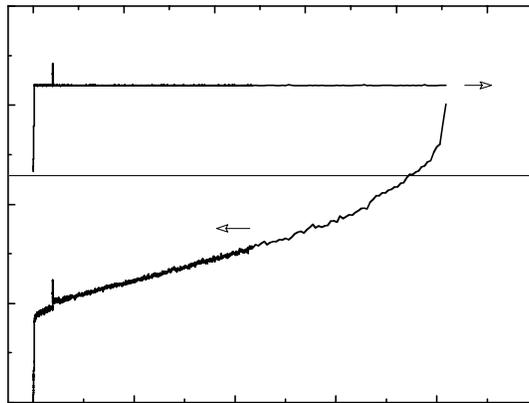


Figure 4. Stress and strain histories for stress-rupture test at 80 MPa and one stress-spike of 20 MPa applied the first hour of the test.

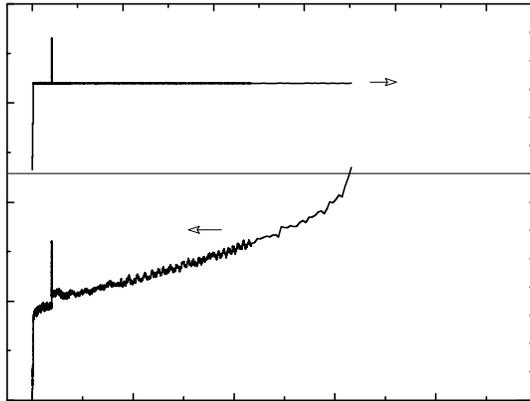


Figure 5. Stress and strain histories for stress-rupture test at 80 MPa and one stress-spike of 40 MPa applied the first hour of the test.

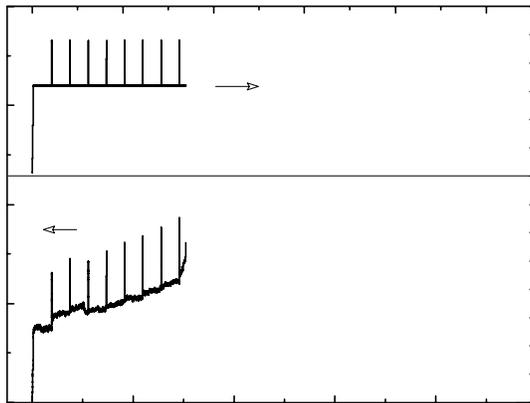


Figure 6. Stress and strain histories for stress-rupture test at 80 MPa and several stress-spike of 40 MPa applied every hour after the first hour of the test.

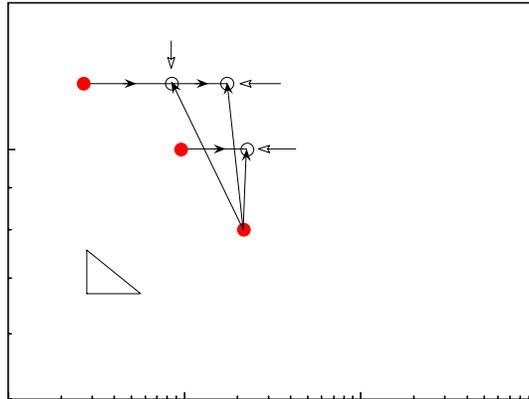


Figure 7. Stress versus time-to-failure for stress-rupture tests at 950°C. Solid circles [8] are from regular stress-rupture tests in ambient air at 950°C. Open circles are from stress-rupture tests that included overstressing conditions.

The open circles in Figure 7 correspond to stress-rupture that included overstressing conditions. Note that the results of these tests are bound by the results of regular stress-rupture tests at 80 and 120 MPa which validates the premise that this test methodology provides a more realistic approach for determining the life of a non-oxide CFCC than would a series of regular stress-rupture tests. In this exercise either one or multiple stress spikes were applied at the first hour of the test and regularly every hour afterwards. In the actual application of the proposed methodology, it will be necessary to evaluate test specimens subjected to random temperature and stress schedules based on the number and the characteristics of transients that are associated with the application of interest. Then, the results from these tests would provide probability of failure and failure rate data necessary to validate life prediction and design schemes and models.

## SUMMARY

A methodology was presented to assess the effect of overstressing on the reliability of CFCCs. The rationale of the methodology is based on the fact that non-oxide CFCCs exhibit distinct behavior at stresses above and below the proportional limit stress, so that regular stress-rupture tests at stresses above and below the proportional limit stress yield either very conservative, or utterly unrealistic results, respectively. The methodology is based on the generation of probability of failure data as a function of random thermomechanical loading histories that would be representative of the transients associated with a given application and that include overstressing events. In this paper the effect of overstressing on the reliability of a CG-Nicalon™/SiC CFCC was investigated. It was found that overstressing reduces the life of the material and that the degree of reduction in life is proportional to the magnitude and frequency of the overstressing events. It was also found that when overstressed, the life of the material is bound by the failure times when subjected to traditional stress-rupture tests at the lowest and largest stresses in the stress history. These results suggest that this methodology appears to be a realistic approach for assessing the reliability of non-oxide CFCCs at elevated temperatures.

## ACKNOWLEDGEMENTS

This work was sponsored by the U.S. Department of Energy, Assistant Secretary for Energy Efficiency and Renewable Energy, Office of Industrial Technologies, Industrial Energy Efficiency Division and Continuous Fiber Ceramic Composites Program, under contract DE-AC05-96OR22464 with Lockheed Martin Energy Research Corp. The author is indebted to Mr. Phil Craig of Allied Signal Composites, Inc. for supplying the specimens used in this study and to his colleagues Arvid E. Pasto, Kristin Breder and Peter F. Tortorelli for reviewing the manuscript.

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