

In-Plane Tensile Properties, Creep Behavior and Failure Mechanisms of CVI and PIP SiC/SiC Composites at Temperatures to 1,650 °C in Air

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Abstract

In-plane tensile and creep properties of 2D woven CVI and PIP SiC/SiC composites with SylramicTMiBN SiC fibers were measured at temperatures to 1,650 °C in air and the data was compared with the literature. Batch-to-batch variations in the tensile and creep properties, and thermal treatment effects on creep, creep parameters, damage mechanisms, and failure modes for these composites were studied. Under the test conditions, the CVI SiC/SiC composites exhibited both matrix and fiber-dominated creep depending on stress, whereas the PIP SiC/SiC composites displayed only fiber-dominated creep. Creep durability in both composite systems is controlled by the most creep resistant phase as well as oxidation of the fibers via cracking matrix. Porosity variations and stress raisers from specimen to specimen caused significant variations in creep behavior and durability. The Larson-Miller parameter and Monkman-Grant relationship were used wherever applicable for analyzing and predicting creep durability. Using a creep model, and the creep parameters of both composites, the magnitude of local stress increase due to porosity variations and stress raisers raisers no creep behavior were determined.

1.0 Introduction

High-strength, creep-resistant SiC fiber-reinforced SiC matrix composites (SiC/SiC) that are microstructurally stable at temperatures to 1,650 °C, are desired for both turbine and hypersonic applications. Currently a variety of SiC fibers are commercially available for ceramic matrix composite (CMC) reinforcement and their thermal and strength stabilities vary at temperatures ranging from 1,300 to 1,800 °C. SiC/SiC composites can be fabricated by hot pressing (HP), silicon melt infiltration (MI), chemical vapor infiltration (CVI), polymer infiltration and pyrolysis (PIP), or a combination of methods (CVI+PIP). The MI SiC/SiC composites are currently being introduced for commercial aircraft turbine engine applications (Ref. 1). The residual silicon present in the matrix of these composites limits their upper use temperature to 1,350 °C. Above this temperature, residual silicon can react with SiC fibers and other constituents, degrading their properties. SiC/SiC composites fabricated by HP are limited to simple shapes and cannot be easily scaled to component fabrication. The SiC/SiC composites fabricated by the other three methods have the potential for high-temperature applications up to 1,650 °C, depending on microstructural stability, strength, and creep and cyclic fatigue resistance of the composite constituents. CVI SiC matrix is known to exhibit high strength and creep resistance at temperatures to 1,500 °C. The BN interface used to obtain crack deflection can be optimized for better oxidation resistance and yet still

provide high crack growth resistance required for long life. For turbine applications, CMCs with a high proportional limit stress (PLS) or initial matrix cracking strength and long-term (>500 hr) creep and cyclic durability are required. The PLS in CMCs is controlled by the modulus of the fibers and matrix, the fiber architecture, the vol% of fiber in the loading direction, the interfacial shear strength between the fibers and the matrix, and the thermal residual stresses in the constituents (Refs. 2 and 3). The PLS can be increased by increasing the vol% of the fibers in the loading direction, the interfacial shear strength between the matrix and the fibers, the matrix fracture toughness and modulus, and by creating compressive residual stress in the matrix by choosing the fiber with greater thermal expansion than the matrix. Whereas, long-term (>500 hr) creep durability of CMCs is controlled by the most creep resistant phase in the composite (Ref. 4). If the creep resistance of the fibers is greater than that of the matrix, stress relaxation occurs in the matrix; even though the load on the composite is the same and the strains in all composite constituents are the same at any time. As a consequence, the stress on the fibers will increase, and correspondingly, the stress on the matrix will decrease with time until initial fracture of the fibers occurs. If the fracture strength range of fibers is significantly lower than that of the matrix, as the stress relaxation occurs in the matrix, the fibers may reach their fracture strength and show multiple fiber cracks before the ultimate failure of the composite. Whereas, if the fracture strength range of the fibers is significantly greater than that of the matrix, as the stress relaxation occurs in the matrix, the fibers can withstand additional stress due to transfer of the load from the matrix until ultimate failure of the composite. In both conditions, the long-term creep and cyclic fatigue durability of the composite is controlled by the fibers (Refs. 4 to 6). On the other hand, if the creep resistance of the matrix is greater than that of the fiber, then the reverse will occur with load transferring to the matrix.

The influence of SiC fiber types, interface coating composition, fiber architecture, and test environments on in-plane tensile, thermal, and creep properties of CVI SiC/SiC composites have been investigated (Refs. 6 to 24). Evans and Weber (Ref. 6) reported increased compliances due to matrix cracking and decreased fiber sliding stresses at 1,200 °C that were almost one order of magnitude smaller than at room temperature. Wilshire et al. (Ref. 20) compared composite creep rates at 1,300 °C to the fiber creep rates to demonstrate the degree of load transfer to the fibers that must occur due to matrix cracking to account for the observed composite creep rates. They observed extensive matrix cracking and pointed out that this cracking facilitates environmental ingress similar to that reported by Henager et al. (Refs. 25 to 27). Zhu et al. (Refs. 7 to 11) conducted creep tests at stresses above the PLS in air and in argon at 1,300 °C on CVI SiC/SiC composites with Hi-NicalonTM and CG-NicalonTM SiC fibers and reported a slower creep rate and longer time to rupture in CVI SiC/SiC composites with Hi-Nicalon[™] SiC fibers, which they attributed to a greater creep resistance of Hi-NicalonTM fibers compared to CG-NicalonTM fibers (Refs. 28 and 29). In addition, they also indicated that creep of bridging fibers at elevated temperatures in inert environments is one possible explanation for the observed creep or slow crack growth of these continuous-fiber composites, although the CVI SiC matrix is more creep resistant than the polymer-derived SiC fibers (Refs. 3, 4, 25 to 30). These results support the hypothesis that creeping fibers transfer stress back to the matrix, causing further matrix cracking, a loss of matrix stiffness, and increased loading of the crack-bridging fibers, ultimately leading to failure. Tressler et al. (Ref. 31) crept CVI SiC/SiC minicomposites with Hi-Nicalon[™] SiC fibers and sintered SiC fibers (also referred to as Carborundum fibers) at 1,200 to 1,400 °C and observed only transient creep both under cracked and uncracked conditions. They also developed a model to predict the creep behavior of the mini composites based on the fiber and matrix creep data generated independently to account for the creep deformation with and without matrix cracks. For the uncracked minicomposites, they used a rule-ofmixtures creep model similar to that used by Holmes et al. (Ref. 5), and for the cracked composites, they used a different approach suitable for a crack-bridging fiber in tension.

Processing and strength properties of PIP SiC/SiC composites with different fiber types have been investigated but only one study examined their creep durability (Ref. 24). In both CVI and PIP SiC/SiC composites, long-term creep durability data are nonexistent in open literature, reliable models to predict long-term creep/cyclic durability accounting for the microstructural inhomogeneity in the matrix have not been developed, the damage/failure mechanisms have not been fully investigated, and the upper use temperature capability of these systems is not known.

The objectives of this study are, from a basic point of view, to determine in-plane tensile properties, creep stress rupture and failure mechanisms of CVI and PIP SiC/SiC composites with SylramicTM-iBN fibers, and from a practical point of view, to determine the upper temperature/stress capability and limitations of these composites for turbine engine and hypersonic applications.

2.0 Experimental Procedure

CVI SiC/SiC composite panels typically having dimensions of 229 mm (L) by 153 mm (W) by 2-2.5 mm (T) were purchased from Hyper-Therm HTC, Inc (currently Rolls-Royce HTC), Huntington Beach, California, USA, and GE Energy-Ceramic Composites Products, LLC, Newark, Delaware, USA. For brevity, these composites are referred to as CVI SiC/SiC (I) and (II). The PIP SiC/SiC composite panels, which had similar dimensions, were purchased from COIC/ATK, San Diego, California, USA. For both types of composites, 2D woven 5 harness satin (5HS) balanced SylramicTM fiber fabric was used as a starting material. However, before the composite fabrication, the required quantity of fabric was heat treated at elevated temperature in N₂ gas to convert SylramicTM to SylramicTM-iBN fibers (Ref. 32). This process creates a thin layer (~100 nm thick) of in situ crystalline BN on the fiber surface that protects the fiber from oxidation and prevents fiber-to-fiber bonding during subsequent fabrication steps. The full descriptions of CVI and PIP SiC/SiC composite fabrication processes are beyond the scope of this paper and have been discussed elsewhere (Refs. 33 to 37). Note that the specific processing conditions are proprietary. Therefore, only salient information of the composite fabrication is described here.

For CVI SiC/SiC composite fabrication, a stack of 8 plies of 2D woven, 0/90 balanced, 5HS fabric of SylramicTM-iBN fibers was tooled and compressed in a graphite fixture to a specific thickness to achieve the desired fiber fraction in the finished composite panel. The whole assembly was transferred into a CVI reactor and infiltrated first with a layer (~0.4 μ m) of BN interface and then with a layer (~2 μ m) of CVI SiC matrix to rigidize the fiber layup. This preform was removed from the tooling and infiltrated with CVI SiC until it could not be infiltrated further. The as-fabricated composite panels contained ~36 vol% SiC fibers, ~6 vol% BN, and ~45 vol% CVI SiC matrix with ~13 vol% closed porosity.

For PIP SiC/SiC composite fabrication, the required size of 2D woven 5HS balanced SylramicTM-iBN fabric was first coated with a thin dual layer (~0.2 μm) of BN/Si₃N₄ coating (also referred to as Mod-1 coating by the vendor) by chemical vapor deposition (CVD) method. The fabric was cut into 153 mm (W) by 229 mm (L) pieces. Each piece of the fabric was coated with a pre-ceramic polymer. Ten pieces of the pre-ceramic polymer-infused 2D woven fabric were stacked, compressed, and pyrolyzed in an autoclave at high temperature in vacuum or in an inert atmosphere to convert the polymer to crystalline SiC matrix. At this stage, the resulting SiC matrix is cracked and porous due to shrinkage of the polymer material as it is converted into crystalline SiC. To improve the density after the initial pyrolysis, the stack is re-infiltrated with pre-ceramic polymer and pyrolyzed. This process of polymer infiltration and pyrolysis is continued until a maximum density is achieved. The as-fabricated composite panels contained ~50 vol% SiC fibers, ~3 vol% BN, and ~32 vol% PIP SiC matrix with ~15 vol% closed porosity.



Figure 1.—Geometry and dimensions of specimen used for tensile and creep testing. (All dimensions are in mm).

The as-processed composite panels were first cut into 153 mm (L) by 13 mm (W) rectangular strips using a diamond tipped cut-off wheel. Each rectangular blank was machined into a dog-bone specimen using a diamond impregnated metal grinder. The CVI SiC/SiC dog-boned specimens were CVI-SiC seal coated following machining, to cover the machined edges with a thin protective layer of SiC. The PIP SiC/SiC dog-boned specimens were re-infiltrated with the pre-ceramic polymer and pyrolyzed to seal the machined edges. The dimensions of the dog-boned specimen are shown in Figure 1. Some CVI SiC/SiC(II) composite specimens were also heat treated at 1,600 °C for 1 hr in flowing argon to examine the influence of heat treatment on in-plane tensile and creep properties of the composites.

Monotonic tensile tests from ambient temperature to 1,650 °C and constant-load tensile creep tests were performed from 1,315 to 1,650 °C in air in an Instron electromechanical series 8562 testing system (Instron Corporation, Norwood, Massachusetts, USA). Room and elevated temperature monotonic tensile tests were conducted at a crosshead displacement rate of 1.27 mm/min. An Instron contact capacitance gage extensometer equipped with bevel-edged SiC rods and 1 percent strain capability was used for measuring displacement in the 25-mm gauge section of the dog-bone specimen. For elevated temperature monotonic tensile tests, both top and bottom grips of the system were water cooled, and the section of the dog-bone specimen between the grips was heated with a MoSi₂ resistance furnace. The displacement in the 25-mm gauge section of the specimen was monitored using a contact extensioneter similar to that used for room-temperature tests. The creep experiments were performed in ambient air in a testing system similar to that used for the high-temperature tensile tests under load control by loading the specimens at 10 N/min to the test load. The specimens were soaked for 30 min at the testing temperature before starting tensile creep tests. Those specimens that survived predetermined exposure time (generally from 100 to 400 hr) were unloaded, and either cooled to ambient or held at the creep testing temperature, and then fast fracture tensile tested to determine their residual in-plane tensile properties. ASTM C1275-18 and ASTM C1337-10 procedures were followed for room-temperature tensile tests and elevated temperature tensile/creep tests, respectively. The tab and fractured regions of the tensile tested specimens were cut for density measurement and fracture surface analysis. Both physical and Archimedes densities were measured at room temperature.

The volume fraction of the fibers in the composite was calculated by dividing the cross-sectional area of the fibers with that of the composite specimen. The cross-sectional area of the fibers was calculated knowing the number of fiber ends per centimeter of the 2D fabric, the average fiber diameter and the number of fibers within the fiber tow, and the number of 2D fabric layers used for composite fabrication. There are 7.87 fiber-tow-ends/cm in a 2D fabric in both longitudinal and transverse directions, and each fiber tow contains 800 filaments. The average diameter of each filament is 10 µm.

Fracture surface analysis of the creep-ruptured and tensile-fractured specimens was conducted in a Hitachi 4700 scanning electron microscope (Hitachi High Technologies Corporation, Tokyo, Japan) to examine topographical features and failure mode(s). To explore damage mechanisms, some creep-ruptured and tensile-fractured specimens were also longitudinally sectioned, mounted in epoxy, and polished on a diamond impregnated metal disc, and then observed in a scanning electron microscope (SEM) or optical microscope. The undamaged portions of the tab region were also mounted, polished and used to examine the as-processed microstructure.

3.0 Results

3.1 Microstructure of CVI and PIP SiC/SiC Composites

Figure 2 to Figure 4 show representative cross-sectional microstructures of CVI SiC/SiC (I) and (II), and PIP SiC/SiC composites, indicating fiber layup, matrix porosity distribution and typical BN interface and CVI SiC coating thickness on SiC fibers. The CVI SiC/SiC (I) composites (Figure 2(a)) appear to be slightly denser compared to the CVI SiC/SiC (II) composites (Figure 2(b)), and they also have a thicker layer of CVI SiC on the surface. A high-magnification photograph of the fiber tow shows the uniformity of the CVI BN interface coating, and the thickness variations of the CVI SiC coating on top of the SiC fibers (Figure 3). The dark ring around the fibers in Figure 3 is the CVI BN coating whose thickness is ~ 0.4 µm. In general, the thickness of CVI SiC coating on top of BN coating within a fiber tow varies and decreases from the outer periphery to the central region of the tow. Comparison of the cross-sectional microstructures within a batch as well as between batches of CVI SiC/SiC composites fabricated by different vendors indicates significant variation in porosity within fiber tows, between fiber tows, and between fiber plies, in addition to variations in thickness of CVI SiC coating. This variation in porosity and nonuniform distribution of CVI SiC within the panel occurs partly due to the randomness of the packing or nesting of the fabric during tooling for CVI fabrication, and partly due to canning of CVI SiC matrix at the surface of the panel and in the interior during the deposition process. On the other hand, the PIP SiC/SiC composite appears to be uniformly dense with micron-scale porosity and matrix microcracks distributed throughout the microstructure (Figure 4(a)). In some regions, fractured fiber tows were also observed, especially in regions where two parallel tows were touching each other. Detailed examination of a SiC fiber/PIP SiC matrix interface region indicates a thin layer (~<0.2 µm) of BN/Si₃N₄ interface coating on top of the fibers (Figure 4(b)).



Figure 2.—Optical photographs of the cross-sections of 2D woven CVI SiC/SiC composites fabricated by different vendors showing fabric layup, porosity variation, and aspect ratio of fiber tows: (a) CVI SiC/SiC (I), (b) CVI SiC/SiC (II).



Figure 3.—High magnification SEM photograph of a cross-section of a 2D woven CVI SiC/SiC composite (I) showing fiber diameter, porosity, CVI BN interface, and CVI SiC coating thickness variation within the fiber tow.



Figure 4.—(a) Optical photograph of a cross-section of a 2D woven PIP SiC/SiC composite showing fabric layup (b) SEM photograph of a fiber tow showing matrix microcracks, porosity, and CVI BN/Si₃N₄ interface coating thickness.

3.2 In-Plane Tensile Behavior and Properties of CVI and PIP SiC/SiC Composites

Room-temperature tensile stress-strain behaviors of as-fabricated CVI SiC/SiC (II) and PIP SiC/SiC composites are shown in Figure 5. The tensile stress-strain behavior of the CVI SiC/SiC (II) composite shows an initial linear region followed by a knee in the curve and another linear region with distinctly different slope until final failure. The slope of the initial linear region is significantly steep, which indicates a high composite elastic modulus. The stress at which the deviation from linearity occurs in the stress-strain curve will from now on be referred to as PLS, and the strain corresponding to PLS will be referred to as the proportional limit strain. In the case of CVI SiC/SiC composites, previous studies have shown that at or near the PLS, a partial through-the-thickness matrix crack is formed (Refs. 3 and 24). As the stress on the specimen is increased, additional through-the-thickness matrix cracks are formed normal to the loading direction and the spacing between the matrix cracks decreases. This process continues until the matrix crack spacing reaches a limiting value. Formation of the initial matrix crack at or slightly above the PLS, formation of multiple matrix cracks as the stress increased above the PLS, and saturation of matrix crack density at ~60 to 70 percent of the ultimate tensile strength (UTS) of the composite have been confirmed from acoustic emission and resistivity data generated during tensile tests of similar composites, as well as by microscopy of specimens loaded to different stress levels until failure (Refs. 3 and 24). Beyond this point, the entire load is carried by the SiC fibers until the ultimate fracture of the composite. This type of composite is classified as a matrix-dominated composite (Ref. 2). On the other hand, the tensile stress-strain behavior of a PIP SiC/SiC composite is parabolic or nonlinear from the beginning until final failure (Figure 5). A pseudo PLS can be estimated but may not represent initial failure of the PIP SiC matrix. In this composite, because the PIP SiC matrix is weak and heavily microcracked due to crystallization and



Figure 5.—Room temperature tensile stress-strain behavior of as-fabricated (a) CVI SiC/SiC (II) and (b) PIP SiC/SiC composites.

shrinkage processes of the multiple PIP cycles performed during fabrication, the stress-strain behavior is essentially controlled by the fibers. Also, the slope of the initial pseudo-linear elastic region is significantly lower than that of CVI SiC/SiC composites. This type of composite is referred to as a fiber-dominated composite (Ref. 3). Whether a composite is matrix or fiber dominated can be easily determined by calculating the effective modulus of the matrix, E_m, using the following equation.

$$E_{\rm m} = (E_{\rm c} - V_{\rm f} E_{\rm f}) / (1 - V_{\rm f}) \tag{1}$$

where E_c is the primary elastic modulus of the composite, E_f is the elastic modulus of the fiber and V_f is the fiber fraction in the loading direction of the composite. Using $E_f \sim 380$ GPa (Refs. 28, 29, 38, and 39) for SylramicTM-iBN fibers, and the composite modulus and half of the total fiber fraction values reported in Table I in Equation (1), the calculated effective moduli values of PIP and CVI SiC matrices are 91 and 230 to 248 GPa, respectively. The significantly lower elastic modulus value of PIP SiC matrix suggests that it is porous/microcracked and bears little load during tensile deformation compared to CVI SiC matrix.

Room-temperature physical and in-plane tensile properties of as-fabricated 2D woven CVI SiC/SiC (I) and (II) composites, and PIP SiC/SiC composites are shown in Table I. The tensile data shown in the table represent an average of 5 specimens, whereas the density and volume fraction data are for an average of 15 to 20 specimens.

Comparison of the data in Table I indicates that the physical densities of all investigated composites are nearly the same. The average elastic modulus value of PIP SiC/SiC composites is statistically lower than those of CVI SiC/SiC composites. The UTS values of CVI SiC/SiC (II) and PIP SiC/SiC composites are nearly the same, although the volume fraction of fibers in the loading direction are significantly different, 18 vol% versus 25 vol%, respectively.

The variation of in-plane tensile properties of CVI SiC/SiC (II) and PIP SiC/SiC composites as a function of temperature is plotted in Figure 6(a) and (b), respectively. The data was generated from the specimens machined from four different composite panels. Each data point in the plots represents an average of 5 specimens and the scatter bands represent \pm one standard deviation. In the as-fabricated condition, both types of composites show significant batch-to-batch variation in in-plane properties, but at elevated temperatures scatter in the data decreases. From ambient temperature to ~1,315 °C, in-plane properties do not degrade significantly, but above this temperature range, all three properties (PLS, E, and UTS) decrease with increase in temperature.

	CVI SiC/SiC (I) composites	CVI SiC/SiC (II) composites	PIP SiC/SiC composites
Fiber layup	2D woven/balanced	2D woven/balanced	2D woven/balanced
Weave	5HS	5HS	5HS
number of plies	8	8	10
Physical density, gm/cc	2.45±0.06	$2.44{\pm}0.05$	2.47±0.01
Archimedes density, gm/cc	Not measured	2.84±0.01	2.65±0.02
Total fiber fraction	0.37±0.01	0.35±0.02	$0.50{\pm}0.02$
Proportional limit stress (PLS), MPa	110±13	128±24	109±12
Proportional limit strain, %	$0.04{\pm}0.01$	$0.05{\pm}0.01$	$0.07{\pm}0.01$
Primary elastic modulus (E), GPa	258±19	272±22	169±19
Ultimate tensile strength (UTS), MPa	440±10	357±75	349±84
Ultimate tensile strain, %	0.49±0.10	0.41±0.11	0.28±0.09

TABLE I.—ROOM- TEMPERATURE PHYSICAL AND IN-PLANE TENSILE PROPERTIES OF AS-FABRICATED 2D WOVEN CVI AND PIP SiC/SiC COMPOSITES WITH SYLRAMICTM-IBN FIBERS





To estimate the maximum fiber stress at failure, the UTS data of both composites shown in Figure 6 were divided by the volume fraction of the fibers in the loading direction for the corresponding composites. For this calculation, it is assumed that UTS is controlled predominantly by the fibers. This assumption is valid because a fully cracked matrix does not bear any significant load. The variation in the estimated fiber strengths with increase in temperature for both composites is shown in Figure 7. It is clear from the figure that the fiber strength retention in the as-processed CVI SiC/SiC (II) composites is better than that in the PIP SiC/SiC composites. This suggests that during processing the fibers in the PIP SiC/SiC composites are degraded to a greater extent than those in the CVI SiC/SiC composites.

Fracture surface morphologies of a CVI SiC/SiC(II) composite tensile tested at room temperature is shown in Figure 8. The fracture surface is irregular and shows limited fiber pull out. Similar features were also observed on the fracture surfaces of the specimens tensile tested at temperatures to 1,650 °C.



Figure 8.—The fracture surface of a CVI SiC/SiC (II) composite tensile tested at room temperature.

Examination of the longitudinal cross sections of CVI SiC/SiC (II) composite specimens tensile tested from ambient temperature to 1,650 °C show similar features: a dominant crack that led to final failure and periodic through-the-thickness matrix cracks. An SEM image of the longitudinal cross section of a CVI SiC/SiC (II) composite specimen tensile tested at 1,650 °C is shown in Figure 9. The dominant crack that led to final failure is on the left in the photograph. The multiple matrix cracks that formed normal to the loading direction (white arrows) during tensile loading can be seen in the photograph. The matrix cracks appear to link internal pores. The average matrix crack spacing was measured from the longitudinal cross sections of tensile tested specimens at different temperatures. Table II shows the measured average matrix crack spacing from 25 to 1,650 °C, which do not show any trend with increase in temperature.

According to composite mechanics, the average matrix crack spacing (S) varies inversely with interfacial shear stress (τ), according to the following equations:

$$\tau = (\sigma A R_f) / (2 S V_f (1 + A))$$
⁽²⁾

$$A = (E_m V_m)/(E_f V_f)$$
(3)

where V_f and V_m are the volume fractions of the fiber and the matrix, respectively, E_f and E_m are the elastic moduli of the fiber and the matrix, respectively, σ is the applied stress at saturation of matrix crack spacing, τ is the interfacial shear strength between the fiber and the matrix, A is a constant, and R_f is the fiber radius.



Figure 9.—The longitudinal cross-section of a CVI SiC/SiC (II) composite tensile tested at 1,650 °C in air showing the dominant fracture path and multiple matrix cracks normal to the loading direction.

Fast fracture tensile test	Average matrix			
temperature,	crack spacing,			
°C	μm			
25	$1,280{\pm}378$			
1,450	1,155±512			
1,550	1,373±471			
1,650	971±106			

TABLE II.—INFLUENCE OF TEMPERATURE ON
AVERAGE MATRIXCRACK SPACING OF
CVI SiC/SiC (II) COMPOSITES

If we assume that the matrix crack spacing value reaches a limiting value between ~60 and 70 percent of the ultimate strength of the composite during a fast fracture tensile test, and that creep effects during the tensile testing are negligible, then one can estimate the interfacial shear strength, knowing S. Using $E_m = 460$ GPa for CVI SiC matrix (Refs. 40), $E_f = 380$ GPa for SylramicTM fibers (Refs. 28, 29, 38, and 39), the fiber and the matrix fractions in the loading direction of 0.18 and 0.45, respectively, the measured matrix crack spacing values shown in Table II, and $R_f = 5 \ \mu m$ for SylramicTM fibers (Refs. 38 and 39) in Equations (2) and (3), variation in the average interfacial shear strength as a function of temperature was calculated. The calculated interfacial shear strength values vary from 21 MPa at room temperature to 14 MPa at 1,650 °C. However, when the scatter in the matrix crack spacing is considered at each temperature, the measured interfacial shear strength values do not seem to show any trend with increase in temperature.

The fracture surfaces of tensile tested PIP SiC/SiC composites from ambient temperature to 1,650 °C are irregular and test temperature did not affect the fracture morphology. An SEM photograph of the representative fracture surface of a PIP SiC/SiC composite tensile tested at ambient temperature is shown in Figure 10. The fracture surface shows no fiber pull out, suggesting high interfacial shear strength between the fiber and the matrix. The longitudinal cross sections of PIP SiC/SiC composites tested from ambient temperature to 1,650 °C also showed only one major crack that caused the final failure. However, in some specimens tensile tested at 1,550 °C and above, a few tunnel cracks were also observed on some 90° tows. Comparison of longitudinal cross-sectional and fracture surface analyses suggest that the damage mechanism(s) and the events that led to the final failure in CVI and PIP SiC/SiC composites are different. In PIP SiC/SiC composites, because of the high interfacial shear strength between the fiber and the matrix, the crack is initiated by a single flaw, possibly a fractured load-bearing fiber tow and the cracked PIP matrix around it, and then the crack grew and linked with similar flaws in different fiber plies causing the final failure. Although not shown, fractured fiber tows have been observed in as-fabricated PIP SiC/SiC composites. In contrast, when CVI SiC/SiC composites are tested under tensile mode, a partial through-the-thickness matrix crack is formed initially around PLS. As the load on the specimen is increased, the matrix crack grows. When it reaches fiber/matrix interface, it gets deflected due to low interfacial shear strength between the fiber and matrix. With increase in load from PLS and UTS, additional matrix cracks are formed at fairly regular intervals perpendicular to the loading direction of the specimen. One of these cracks becomes a dominant crack and linked with other cracks in the same plane causing the final failure.



Figure 10.—The fracture surface of a PIP SiC/SiC composite tensile tested at room temperature.

3.3 Creep Properties of CVI SiC/SiC Composites

Tensile creep tests were conducted on CVI SiC/SiC (I) and (II) composites at stress levels above and below the PLS. The creep stresses were chosen based on the tensile test data shown in Figure 6. From ambient temperature up to 1,315 °C, the PLS remains nearly the same, but above this temperature it decreases with increase in temperature: the PLS at room temperature is ~120 MPa and decreases to ~89 MPa at 1,650 °C. Above the PLS, the CVI SiC matrix cracks during initial creep loading, but below the PLS, the matrix may or may not crack during the creep test, depending on the local stress.

During the course of this investigation, it was recognized that creep resistance of CVI SiC/SiC composites can be significantly improved by thermal heat treatment above 1,450 °C in argon or vacuum. However, the thermal heat treatment also causes a small loss in PLS as well as E, but UTS remains the same. To investigate the influence of thermal heat treatment on creep durability, some of the CVI SiC/SiC composite speimens were also thermally heat treated at 1,600 °C in argon for 1 hr.

The influence of stress on the creep behavior at 1,315 °C is shown in Figure 11. The creep data shown in the figure were all generated from the same panel. At 155 MPa, the specimen failed after 147 hr (the downward arrow in the figure indicates failure), but at lower stresses specimens survived 300 hr exposure. According to the figure, the accumulated creep strain increases with increase in creep stress at a fixed exposure time. However, batch-to-batch as well as within batch variations in creep behaviors do exist even at a fixed temperature and stress because of the local variations in microstructure and porosity in CVI SiC/SiC composites. These variations will also create a nonuniform strain field, and result in a greater amount of strain within the gauge section of the specimen when compared with the gauge section in which both the stress and strain fields are uniform. In addition, if these local stresses are greater than the PLS, the CVI SiC matrix will crack locally either during the initial creep loading or during the creep deformation, exposing bridging SiC fibers for oxidation and crack growth.

To determine variability in creep behavior at a fixed temperature and stress, creep tests were conducted on specimens from different batches of CVI SiC/SiC composites. Figure 12(a) and (b) show typical variation in creep behavior seen at 1,450 °C at 69 MPa from different batches of CVI SiC/SiC composite fabricated by two different vendors. Although observed but not shown is that at a fixed temperature, variability in creep behavior decreases with decrease in creep stress, and conversely at a fixed stress, variability in creep behavior decreases with decrease in temperature. Also shown in Figure 12(b) is the influence of thermal heat treatment on the creep behavior of CVI SiC/SiC (II) composites generated from the same panel. In general, the creep curves in Figure 11 and Figure 12 display an instantaneous elastic strain upon application of the load followed by two or three, time dependent creep regimes: a primary, a pseudo-secondary and, rarely, a tertiary creep. The elastic strain is recoverable. The accumulated creep strain is partially recoverable with time if unloaded and held at the creeping temperature. The primary and secondary creep regimes follow a power law.

In Figure 11, at high stresses ($\sim \geq 138$ MPa), the CVI SiC matrix may be partially or completely cracked. If completely cracked, the SiC fibers control the creep rupture life. If the CVI SiC matrix is partially cracked, both the SiC fibers and the CVI SiC matrix control the creep rupture life. In both cases, oxidation of the exposed fibers between the segments of fully cracked or partially cracked CVI matrix will reduce creep rupture life. At stresses below the PLS, the CVI matrix may crack during creep deformation due to stress relaxation in the fiber, and the stress on the matrix will increase with time and eventually reach its fracture strength. Here, creep rupture life is controlled by the slow crack growth rates of CVI SiC matrix and SiC fibers. At stresses well below the PLS, such as at 69 MPa in Figure 11, creep tests will last a long time (≥ 500 hr). Although stress relaxation is occurring in SiC fibers under these conditions, the CVI matrix can withstand the increase in stress without considerable damage or failure.



Figure 11.—Influence of stress on tensile creep behavior of CVI SiC/SiC (I) composites tested at 1,315 °C.

The thermal heat treatment does show improved creep resistance of CVI SiC/SiC composites (Figure 12(b)) as long as creep tests were conducted at stresses below the PLS and the CVI SiC matrix was not cracked. Although not shown, above the PLS, creep resistance of heat-treated composites is similar to that of the non-heat-treated composites.

Further examination of the creep curves indicates that a steady-state creep rate may not be reached in CVI SiC/SiC composites at a constant temperature and stress, even when the creep tests were continued for >400 hr. To illustrate this, the variation of apparent minimum creep rate with exposure time for the specimens crept at 1,450 °C at 69 MPa for up to 500 hr is shown in Figure 13. In this figure, the apparent creep rates of some specimens that failed during the creep tests and some specimens that were tested for 100, 200, and 500 hr without failure are shown.

It is clear from the figure that the apparent creep rate decreases with increase in time without reaching a steady state even after 500 hr of testing. Decreasing apparent minimum creep rate with increasing exposure time is due to two reasons: first, CVI SiC matrix itself does not show a steady-state creep. This fact is substantiated by the creep data of DiCarlo et al. (Refs. 41 and 42) on CVD SiC fibers and Tressler et al. (Ref. 43) on CVI SiC/SiC mini composites. Second, stress relaxation occurs due to difference in the creep resistances of polymer-derived SiC fibers and CVI SiC matrix. The estimated or reported values of apparent steady-state creep rate for polymer-derived SiC fibers such as SylramicTM-iBN and Hi-Nicalon- S^{TM} and CVI SiC matrix are ~10⁻⁷ to 10⁻⁸/sec (Refs. 28 and 29) and 10⁻⁹ to 10⁻¹⁰/sec (Ref. 30), respectively, at 1,400 °C and at stresses up to 275 MPa. Since the creep resistance of polymer-derived SiC fibers is an order-of-magnitude lower than that of the CVI SiC matrix, stress relaxation occurs in the SiC fibers during a creep test. As a consequence, the load on the fibers decreases and that on the CVI SiC matrix increases with increase in exposure time until the final failure of the composite. Therefore, longterm creep durability of CVI SiC/SiC composites below the matrix cracking stress is controlled by the CVI SiC matrix and not by the polymer-derived SiC fibers. Additionally, not knowing a steady-state minimum creep rate in this system will also cause substantial error in determining the creep parameters such as the primary and secondary stress exponents and other creep constants. Therefore, to reduce uncertainty in determining the creep parameters, long-term (>300 hr) creep tests should be conducted.



Figure 12.—Variation in tensile creep behavior of CVI SiC/SiC composites at 1,450 °C and at 69 MPa: (a) CVI SiC/SiC (I), (b) CVI SiC/SiC (II).



Figure 13.—Variation of apparent minimum creep rate with exposure time for CVI SiC/SiC (II) composites crept at 1,450 °C at 69 MPa. The symbols with an arrow indicate run-out.

When the creep curves shown in Figure 11 and Figure 12 were replotted as tensile creep strain (after deducting the initial elastic strain) with exposure time, $t^{(1/3)}$ power, a linear relationship was observed as shown in Figure 14(a) and (b). This relationship is seen only when CVI SiC matrix is controlling the creep. Also, note that the slope of the tensile creep strain versus $t^{(1/3)}$ increases with increase in creep stress at a fixed temperature. The slope also increases with increase in temperature (see Figure 15).

The variation in apparent minimum creep rate with stress is shown in Figure 16 for CVI SiC/SiC (I) and (II) composites creep tested from 1,315 to 1,550 °C. For this plot the apparent minimum creep rate was measured at 300 hr from the creep data of the specimens that lasted 300 to 400 hr. In this figure at each temperature between 1,315 and 1,550 °C, ln(apparent creep rate) varies linearly with ln(tensile creep stress), and the slope of the line, which is the stress exponent of creep, is ~1, but the intercept of the line on y-axis at each temperature is slightly different.













Previous creep studies on macro- and mini-CVI SiC/SiC composites have reported stress exponents varying from 1 to 25 (Refs. 7, 22, 31, and 43). Shinavski (Ref. 22) reported stress exponent values between 4 and 13 at temperatures from 1,482 to 1,600 °C at stress values between 69 and 138 MPa for CVI SiC/SiC composites with SylramicTM-iBN fibers. Zhu et al. (Ref. 7) reported stress exponent values between 5 and 25 at temperatures from 1,000 and 1,300 °C at stress values between 30 and 200 MPa for CVI SiC/SiC composites with Hi-NicalonTM fibers. Tressler et al. (Refs. 31 and 43) reported a stress exponent value of 1.9 for CVI SiC matrix. In the ceramic literature, a stress exponent of 1 typically indicates diffusion creep. Although the reasons for such wide variations in stress exponents in CVI SiC/SiC composites are not known, it is speculated that uncertainty in calculating the apparent minimum creep rate or different creep mechanisms operating under the tested conditions are some of the causes. If the CVI SiC matrix is completely cracked and does not carry any significant load, the apparent creep rate is controlled by the fibers. However, oxidation of the fibers between the cracked matrix segments can change the apparent creep rate with time. If the CVI SiC is partially cracked, then the apparent creep rate is controlled by both the fibers and the matrix. The extent of cracking during the creep deformation, the slow crack growth rate of CVI SiC matrix and the fiber and oxidation of the fibers in the cracked matrix regions, will influence the apparent creep rate. If the CVI SiC matrix is uncracked, uncertainty in the apparent creep rate will arise if creep tests were not conducted for long enough time since the apparent creep rate decreases as indicated in Figure 13.

With the knowledge of the apparent creep rates at 300 hr for CVI SiC/SiC (I) and (II) composites tested at 69 MPa from 1,400 to 1,550 °C, we determined the activation energy for primary creep (Q) by plotting ln(apparent creep rate) versus 1/T (Figure 17). This plot yields a linear behavior. The slope of which is -Q/R. Knowing the slope of the line and the gas constant R = 8.3144 J/mol K, Q was calculated. The calculated value of Q is 619 kJ/mol. Creep activation energy varying from 400 to 900 kJ/mol has been reported for CVD or CVI SiC matrix (Refs. 41, 43, and 44), and the value reported here is well within that range. The observed wide variation in creep activation energy is probably due to subtle variation in the stoichiometry of CVD/CVI SiC fabricated by different vendors as well as different creep testing methods used (tensile, compression, bend stress relaxation, etc.).



Figure 17.—Variation of apparent creep rate with reciprocal of absolute temperature for CVI SiC/SiC (I) and (II) composites creep tested from 1,400 to 1,550 °C at 69 MPa.

The morphology of the fracture surface and fracture path of a creep ruptured CVI SiC/SiC composite specimen is shown in Figure 18. The fracture surface indicates no significant fiber pull-out, and a relatively flat region up to the red dotted line on the right side of the specimen. The energy dispersive spectroscopy (EDS) examination of this region indicates formation of silica scale. The dominant crack that caused creep rupture probably initiated and propagated from this region. The longitudinal cross section of the same specimen near the fractured end shows a single dominant crack causing the stress rupture and no additional secondary through-the-thickness matrix cracks normal to the loading direction are noticed.

Creep durability of CVI SiC/SiC (I) and (II) composites with SylramicTM-iBN fibers at 1,315 and 1,450 °C is plotted in Figure 19 and compared with the literature data on other CVI SiC/SiC composites with CG NicalonTM, Hi-NicalonTM, and SylramicTM-iBN fibers. The CVI SiC/SiC composites containing CG NicalonTM and Hi-NicalonTM fibers had a carbon interface, and the CVI SiC/SiC (I) and (II) composites and the composite from Reference 22 had a BN interface and SylramicTM-iBN fibers. Comparison of the plots in Figure 19(a) indicates that creep durability of CG NicalonTM and Hi-NicalonTM fibers was significantly lower than that containing SylramicTM-iBN fibers predominantly due to relative creep resistance of the fibers; creep resistance of SylramicTM-iBN > Hi-NicalonTM fibers. Wide variation in creep rupture life was seen at both temperatures, even at a fixed stress. In Figure 19(b), the creep life of CVI SiC/SiC composite (I) appears to be better than that of CVI SiC/SiC composite (II); possibly due to higher PLS in the former (denser material). The arrows in Figure 19(b) represent specimens crept for predetermined exposure times, where a run out condition was achieved.



Figure 18.—The fracture surface of a CVI SiC/SiC (II) composite specimen tensile creep ruptured at 1,450 °C at 69 MPa for 58 hr.



Figure 19.—Creep durability of CVI SiC/SiC composites at (a) 1,315 °C, and (b) 1,450 °C.

One method used for estimating and comparing creep rupture lives of different CMC materials is by the Larson-Miller parameter (LMP). This parameter relates creep rupture time with creep stress and temperature.

$$LMP = T(\log(t_R) + D)$$
(4)

where T is the temperature (K), t_R is the creep rupture time and D is a constant. For SiC fibers and SiC/SiC composite systems, the best fit value of D is 22 (Ref. 41). One basic assumption in LMP is that a single creep mechanism operates under all tested conditions and is responsible for creep rupture. Figure 20 shows the LMP plot for CVI SiC/SiC (I) and (II) composites. Included in the plot LMP value for the heat-treated CVI SiC/SiC (II) composite. Also shown in the figure for comparison are the LMP plots published in the literature for the 2D woven CVI SiC/SiC composites with CG-NicalonTM and Hi-NicalonTM SiC fibers with a CVI carbon interface (Ref. 10) and SylramicTM-iBN fibers with a CVI BN interface coating (Ref. 22). In all these composites, the total vol% of SiC fibers is nearly the same (\sim 36 vol%) but the density values and interface coatings are different. It is apparent in Figure 20 that CVI SiC/SiC composites with different SiC fiber types, and the composites with the same type of fibers but with different densities, follow different LMP plots. Generally, as the matrix density increases, the LMP plot shifts to the right of the diagram, and as the creep resistance of the fiber or the interfacial shear strength between the fiber and matrix decreases (as in the case of the fibers coated with carbon interface), the LMP plot shifts to the left of the diagram. In the figure, the LMP plot of CVI SiC/SiC composites with CG-NicalonTM and Hi-NicalonTM SiC fibers show significantly lower creep rupture life compared to those containing SylramicTM-iBN fibers because creep rates of CG-NicalonTM and Hi-NicalonTM SiC fibers are an order of magnitude greater than that of SylramicTM-iBN fibers under the test conditions (Refs. 28 and 29). Another noteworthy observation in the plot is that creep durability of nonheat-treated and heat-treated CVI SiC/SiC (II) composites is nearly the same, which indicates that both non heat-treated and heat-treated composites were cracked either during initial loading or during creep.

A limitation of LMP plots of SiC/SiC CMCs is that no single creep mechanism operates throughout the entire range of stress, temperature, and exposure conditions. The creep controlling mechanism will invariably vary depending on the stress, temperature, matrix crack density, and oxidation of constituents. At high stresses (>60 to 70 percent of the UTS), since the CVI SiC matrix is completely microcracked and can carry minimal load, creep rupture of the CMC is predominantly controlled by the fibers. Oxidation of the exposed fibers between the segments of microcracked CVI matrix may affect creep

rupture time. In the intermediate stress range (between 103 and 180 MPa), the CVI SiC matrix may be partially cracked as soon as the load is applied or during a creep test. In this stress range, the CVI SiC/SiC composites with greater bulk density showed slightly improved creep rupture life, possibly due to higher matrix cracking stress. Creep rupture time in this regime is controlled by the CVI SiC matrix, the SiC fiber, and oxidation of the bridged fibers between cracked matrix blocks. Although the LMP plot is controlled by different constituents at different stresses and temperatures and the extent of fiber oxidation rates, it can still be used qualitatively to compare the creep durability of different composite systems as well as to understand the influence of fiber creep, oxidation and matrix density effects.

The specimens that survived predetermined creep exposures were unloaded from the creep temperature, and then fast fracture tensile tested either at the creep test temperature or at room temperature to determine the influence of accumulated non-recoverable creep strain on the tensile stress-strain behavior and on inplane tensile properties. Figure 21(a) and (b) show the tensile stress-strain behaviors of as-fabricated CVI SiC/SiC(II) composites at 1,450 and 1,550 °C and those of crept composite at 1,450 and 1,550 °C at 69 MPa for 100 and 400 hr, but tested at the creep testing temperatures. Comparison of tensile stress-strain behaviors in Figure 21(b) indicates no significant effect of accumulated creep strain on the tensile strain behavior and that PLS (stress corresponding to deviation from linearity) and E (primary elastic modulus) of as-fabricated and crept composites are nearly the same. Whereas, UTSs of the crept composites at 1,550 °C are 10 to 20 percent lower than those of as-fabricated composites.

Residual in-plane tensile properties of crept CVI SiC/SiC composite specimens that survived predetermined exposures are shown in Table III. For comparison, in-plane tensile properties of as-fabricated CVI SiC/SiC composites tested at ambient temperature as well as the creep test temperatures are also shown. No significant loss in in-plane tensile properties was observed in crept composites when compared to those of as-fabricated composites tested at the same temperature. This suggests that accumulated creep strain within certain limits has no significant effect on the in-plane tensile properties of this composite. This is expected in composites in which the matrix is uncracked and controls the creep behavior and rupture life. However, beyond certain limits, the accumulated creep strain will cause slow crack growth, cavitation, and adverse residual stresses in the composite matrix, degrading the in-plane tensile properties.



Figure 20.—Larson-Miller plot for CVI SiC/SiC composites.



Figure 21.—Comparison of fast fracture tensile stress-strain curves of CVI SiC/SiC (II) composite before and after creep testing: (a) 1,450 °C (b) 1,55 0°C.

Specimen history (number of specimens tested)	Tensile test temperature, °C	Proportional limit stress (PLS), MPa	Proportional limit strain, %	Primary elastic modulus (E), GPa	Ultimate tensile strength (UTS), MPa	Ultimate tensile strain, %
As fabricated (12)	25	128±24	0.05±0.01	272±22	357±75	0.41±0.11
Crept at 1,315 °C at 138 MPa for 250 hr (2)	25	124±4	0.05	243±14	353±32	0.31±0.02
As fabricated (3)	1,450	101±16	0.04±0.01	224±17	295±36	0.46±0.01
Crept at 1,450 °C at 69 MPa for 100 hr (4)	1,450	102±7	0.05±0.01	227±18	246±50	0.41±0.14
Crept at 1,450 °C at 69 MPa for 500 hr (3)	1,450	117±15	0.05±0.02	240±52	303±50	0.50±0.04
As fabricated (6)	1,550	87±10	0.05±0.01	198±18	239±26	0.45±0.08
Crept at 1,550 °C at 35 MPa for 100 hr (2)	1,550	94±2	0.05	163±7	167±20	0.27±0.05
Crept at 1,550 °C at 69 MPa for 100 hr	1,550	95	0.04	238	209	0.34
As fabricated	1,600	45	0.02	199	87	0.11
Crept at 1,600 °C at 35 MPa for 100 hr (3)	1,600	83±14	0.05±0.02	216±9	147±5	0.22±0.06
As fabricated (3)	1,650	89±5	0.06±0.1	154±12	182±10	0.59±0.04
Crept at 1,650 °C at 35 MPa for 100 hr	1,650	66	0.05	140	118	0.13

TABLE III.—RESIDUAL IN-PLANE TENSILE PROPERTIES OF CREPT CVI SiC/SiC (II) COMPOSITES

Fracture surface analysis and observation of the longitudinal section of crept and then tensilefractured specimens showed features similar to as-received specimens tensile tested at the same temperature: highly irregular fracture surface and multiple thorough-the-thickness matrix cracks normal to the loading direction.

To understand the influence of accumulated creep on interfacial shear strength, matrix crack spacing measurements were conducted on two specimens tensile fractured at 1,450 °C following creep testing at 1,450 °C at 69 MPa for 100 hr. The average matrix crack spacing in the first specimen is $716 \pm 85 \,\mu\text{m}$ and the other is $822 \pm 294 \,\mu\text{m}$. These values are well within the scatter band of matrix crack spacing

values shown in Table II, which suggests that interfacial shear strength of as fabricated and crept/tensile tested composites are nearly the same.

3.4 Creep of PIP SiC/SiC Composites

Figure 22(a) and (b) show the effect of stress on creep behavior at a fixed temperature, and typical variability seen in creep behaviors at a fixed stress of PIP SiC/SiC composites at 1,315 and 1,450 °C. At 1,315 °C, variability in creep behaviors at constant stress is relatively low; the accumulated creep strain increased with increase in stress at a fixed exposure time. On the other hand, at 1,450 °C and above, variability in creep behavior is so large that it is difficult to determine stress effects on the creep behavior. In general, a typical creep curve of these composites exhibits three separate regimes: an initial elastic regime after application of the load, followed by a short time dependent primary creep, and then an extended linear region that lasted until specimen failure. At low stresses and temperatures, the primary creep region lasted a long time (up to 100 hr). At 1,315 °C, the composites survived 200-hr creep tests at stresses up to 172 MPa, but at 1,450 °C and higher, the composites lasted less than 100 hr at stresses >103 MPa. At 1,650 °C, the composites do not show 100-hr creep durability even at low stresses.

Because of the short duration of primary compared to secondary creep in this system, only the creep parameters of the latter were investigated. To determine the creep stress exponent (n') and activation energy (Q) for the secondary creep, the measured minimum creep rates at a fixed temperature and different stresses, and those at a fixed stress and at different temperatures were plotted. The slope of ln(minimum creep rate) versus ln(tensile creep stress) and that of ln(minimum creep rate) versus 1/T plots, yield the stress exponent (n') and –Q/R, respectively. Knowing the slope and R, Q was calculated. The plots are shown in Figure 23 and Figure 24. These plots indicate that between 1,315 and 1,550 °C, the calculated creep stress exponent is ~4, and the activation energy for the secondary creep is ~913 kJ/mol. If we assume that PIP SiC/SiC composites exhibit fiber-dominated creep, then we would expect the creep stress exponent (n') to resemble that of SiC fibers, which is between 1 and 3 (Refs. 28 and 45). A higher value of creep exponent (~4) in these composites is due to local stress being much greater than the global applied stress, and the error in knowing true stress. A high value of activation energy implies that creep is controlled by grain boundary diffusion similar to individual SiC fiber filaments (Refs. 45 and 46).



Figure 22.—Influence of stress on creep behavior of PIP SiC/SiC composites at (a) 1,315 °C and (b) 1,450 °C.







Figure 24.—Variation of minimum creep rate with reciprocal of absolute temperature for PIP SiC/SiC composites creep tested from 1,315 to 1,550 °C at 103 MPa.

The fact that PIP SiC matrix is weak, microcracked, and contains microporosity, and is susceptible to tunnel cracks in the 90° tows, indicates that it may not bear significant load, and that creep deformation in this composite is controlled predominantly by the fibers. Because fiber creep is the only dominant creep mechanism operating under the test conditions, the LMP plot and Monkman-Grant relationship can be used for predicting creep durability.

The LMP plot of the creep rupture data of PIP SiC/SiC composites is shown in Figure 25. At each stress, significant scatter in the data exists.

The Monkman-Grant relationship relates steady-state creep rate to creep rupture time according to the equation.

$$\varepsilon^{*M} t_{R} = K, \tag{5}$$

where ϵ^{*M} is the steady-state creep rate, t_R is the creep rupture time, M and K are constants. The Monkman-Grant plots for PIP SiC/SiC composites tested at 1,315 and 1,450 °C are shown in Figure 26. The slope of each line shown in the plot is the M constant, which is ~1 at both temperatures.

Examination of the fracture surfaces of creep ruptured specimens from 1,315 to 1,650 °C at different stresses showed features similar to that found on as-fabricated specimens tensile tested at room temperature: fracture occurring in different fiber plies and linking, and no fiber pull out. The longitudinal cross sections of the same specimens near the fractured end indicate macro-cracks in [90°] fiber tows normal to the loading direction. However, the density of these cracks increases with increase in the temperature at a fixed stress and increase in stress at a fixed temperature. Figure 27 shows a longitudinal cross section of a PIP SiC/SiC composite creep ruptured at 1,450 °C at 69 MPa after 351 hr exposure. In the figure, the dominant crack that led to final failure as well as random tunnel cracks that developed during creep deformation on 90° tows can be seen.



Figure 26.—Monkman-Grant plots for PIP SiC/SiC composites.



Figure 27.—The longitudinal cross section of a PIP SiC/SiC composite creep ruptured after 351 hr exposure at 1,450 °C at 69 MPa showing the path of the dominant crack that caused failure and formation of tunnel cracks on 90° tows.



Figure 28.—Comparison of tensile stress-strain behaviors of PIP SiC/SiC composites before and after creep.

Figure 28 shows the tensile stress-strain behaviors at 1,450 °C for the specimens that were previously creep tested at 69 MPa at 1,450 °C for 100 and 335 hr, as well as the tensile stress-strain behavior of the as-fabricated composite at 1,450 °C. Comparison of the tensile stress-strain behaviors indicates that the UTSs of some crept composites were lower than those of as-fabricated composites tested, but there is no trend with exposure time. The residual in-plane tensile properties of all crept specimens are shown in Table IV. In general, in-plane tensile properties of the composites tested at 1,315 °C, but those composites crept at 1,450 °C and above do show some loss in UTS and ultimate tensile strain with increase in exposure time because of creep and oxidation effects on SiC fibers.

Maximum temperature and stress at which 2D woven CVI and PIP SiC/SiC composites reinforced with SylramicTM-iBN fibers can be used for 100- and 500-hr applications have been estimated based on the creep data generated in this study. The 100- and 500-hr durability are typically projected for

hypersonic and turbine engine applications, respectively. It is assumed here that for specific applications, these composites will need surface coating(s) to protect them against environmental attack. Figure 29 shows the plots. In both composites, creep durability decreases with increase in temperature as well as increase in stress.

Specimen history (number of specimens tested)	Tensile test temperature, °C	Proportional limit stress (PLS), MPa	Proportional limit strain, %	Primary elastic modulus (E), GPa	Ultimate tensile strength (UTS), MPa	Ultimate tensile strain, %
As fabricated (13)	25	109±12	0.07 ± 0.01	169±19	349±84	0.28±0.09
As fabricated	1,315	98	0.06	182	250	0.23
Crept at 1,315 °C at 103 MPa for 200 hr	1,315	111	0.08	148	256	0.21
Crept at 1,315 °C at 172 MPa for 200 hr	1,315	112	0.08	143	255	0.21
As fabricated (3)	1,450	106±12	0.09±0.01	118±2	293±16	0.31±0.02
Crept at 1,450 °C at 69 MPa for 100 hr (4)	1,450	106±4	0.08±0.01	121±21	246±32	0.24±0.03
Crept at 1,450 °C at 69 MPa for 500 hr (2)	1,450	93±24	0.06±0.02	149±8	227±50	0.20±0.03
Crept at 1,450 °C at 103 MPa for 200 hr	1,450	101	0.06	166	156	0.10
As fabricated (6)	1,550	90±17	0.09±0.01	104±11	235±17	0.29±0.03
Crept at 1,500 °C at 69 MPa for 100 hr (2)	1,550	107±3	0.11±0.01	94±15	195±28	0.21±0.05

TABLE IV.—RESIDUAL IN-PLANE TENSILE PROPERTIES OF CREPT PIP SiC/SiC COMPOSITES



Figure 29.—Variation of 100- and 500-hr creep durability with temperature for (a) CVI SiC/SiC and (b) PIP SiC/SiC composites.

3.5 Creep Model

In general, a creep curve of a fiber-reinforced ceramic matrix composite exhibits three distinct regions: an elastic strain region after immediate application of load, a primary creep region where strain rate decreases with increase in exposure time, followed by a region where creep rate remains relatively constant with increase in exposure time. Out of the total accumulated strain, the elastic strain is fully recoverable upon unloading. In the literature, various empirical equations have been developed to describe and model the first three regions. One such empirical relationship that relates accumulated creep strain with time relation is:

$$\varepsilon_{\rm AC} = \varepsilon_{\rm i} + ({\rm A}\sigma^{\rm n}(1 - \exp(\beta t)) + {\rm B}\sigma^{\rm n'}t \tag{6}$$

where ε_{AC} and ε_i are the accumulated creep strain, and the initial elastic strain in the composite, respectively, σ is the composite stress, and A, n, n', B, and β are the empirical constants. The activation energies for primary and secondary creep are included in these constants. The model assumes uncracked CMC matrix, same strain rate among composite constituents and perfect load transfer across the interface and does not account for stress relaxation effects.

As described earlier, at any fixed temperature and stress, the accumulated creep strain with exposure time plot of CVI SiC/SiC composites exhibits instantaneous elastic strain and primary creep regions. In addition, the experimental data indicate that the creep rate in primary creep regime is dependent not only on temperature, but also on time, and does not reach a minimum creep rate even after a prolonged exposure. If we consider primary creep is also a thermally activated process with an activation energy, then the conventional analysis that measures the exponential dependency of the creep rate on temperature alone should be modified to include time dependency, although time and temperature are both considered to be independent variables. Such modifications have been suggested in the literature to analyze primary creep (Refs. 41, 43, 47, and 48). Therefore, Equation (6) can be modified after deducting the elastic strain from the total strain to a simple power law relating accumulated creep strain (ϵ_{AC}) to applied stress, exposure time (t) and temperature (T).

$$\varepsilon_{\rm AC} = [A] [\sigma^{\rm n}][t^{\rm p}][e^{(-pQ/RT)}]$$
⁽⁷⁾

$$\varepsilon_{\rm AC} = [C][t^p] \tag{8}$$

where $C = [A][\sigma^n][e^{(-pQ/RT)]}]$, p is the time exponent, Q is the activation energy for primary creep, R is the gas constant, T is absolute temperature and all other constants are as defined earlier. Differentiation of the Equation (8) with time, t, yields,

$$\epsilon^*_{AC} = [A] [\sigma^n] [pt^{(p-1)}] [e^{(-pQ/RT)}]$$
(9)

Taking natural log on both sides

$$\ln(\epsilon^*_{AC}) = \ln[A] + n\ln[\sigma] + \ln[pt^{(p-1)}] + \ln[-pQ/RT]$$
(10)

The constants in Equations (7) to (10) can be determined from a series of isothermal and isostress creep tests.

All the creep data generated on CVI SiC/SiC composites in this study indicate that accumulated creep strain varies linearly with $t^{(1/3)}$ in the primary creep region. Therefore, in Equation (8), the creep time constant, p, is 1/3. Knowing p, the value of constant C required to fit the experimental creep curve was determined. With the knowledge of p and C, then creep rate at a specific time (for example 300 hr) was

calculated using Equation (9). Using Equation (10), other constants were determined. At a constant temperature, 1st, 3rd and 4th terms, and at a constant stress, 1st, 2nd and 3rd terms in Equation (10) are constants. By plotting ln(creep rate) versus ln(creep stress) at a constant temperature, and ln(creep rate) versus ln(1/T) at constant stress, the primary creep exponent (n) and the apparent activation energy (Q), respectively were determined. The slope of the first plot yields, n and that of the second plot yields, Q. The calculated primary creep parameters for CVI SiC/SiC (I) and (II) composites with SylramicTM-iBN fibers from 1,315 to 1,550 °C are shown in Table V. Results show that under the testing conditions, the time and stress exponents and the activation energy remained the same for CVI SiC/SiC composites fabricated by two different vendors, but constant A is different. However, within a vendor lot, constant A remains relatively constant up to 1,482 °C. Above this temperature, constant A increased. One reason for such a difference in constant A from vendor to vendor is possibly due to subtle differences in the composition and microstructure of the CVI SiC matrix.

Although the creep time exponent, p, is the same for all CVI SiC composites tested in this study, significant variations in the creep behaviors were observed when multiple specimens were tested at a fixed temperature and stress from the same batch and/or from different batches of CVI SiC/SiC composites fabricated either from the same vendor or from different vendors. These variations are possibly caused by average local stress being different from the global applied stress. Note that all creep tests were conducted under load control, and the global stress was calculated based on outer dimensions of the specimen and the load, but in reality, net load bearing cross-sectional area varies from region to region in CVI SiC/SiC composites because of pore size variation. Reduced cross section increases average stress on the specimen. In addition, the presence of stress raiser (bent fiber tow during weaving), sharp corners of pores, and tooling bumps (CVI SiC nodules on the specimen surface) will further enhance the local stresses.

To estimate the magnitude of the average local stress increase compared to the global applied stress, and the influence of average local stress increase on the creep behavior, the creep parameters shown in Table V were used to determine the stress at which the predicted creep behavior matches the experimentally determined behavior. To do this, the exponential term in Equation (7) was calculated first with the knowledge of p, Q, T, and R, and then multiplying it with the creep constant A for the temperature of interest. The product is multiplied with a value of stress to match the value of C measured for the experimentally generated creep behavior at a constant temperature and stress. Knowing C, time t is arbitrarily varied in Equation (7) to calculate strain. The ensuing predicted behavior is compared with the experimentally generated behavior. Figure 30(a) and (b) shows observed creep behaviors based on global (applied) stress and predicted average local stress for the same specimen for CVI (I) and (II) SiC/SiC composites tested at 1,315 and 1,450 °C. The data shown in Figure 30(a) and (b) indicate that local stresses could be 2 to 3 times greater than the global stress. Another conclusion that can be drawn from the data is that when the local stress exceeds a certain value, the specimen fails during the creep test as indicated in the figure by the downward arrows. Although, the stress range within which a matrix crack initiates and propagates is not fully understood, the minimum local stress required to initiate and propagate matrix cracks appears to decrease with increase in temperature: (~300 MPa at 1,315 °C versus ~200 MPa at 1,450 °C). If net load bearing cross-sectional area variation and stress raisers within the gauge section of the specimen can be mapped by nondestructive or destructive techniques, then by using a finite element model it could be possible to separate the contributions of porosity and stress raisers on local stresses as well as to predict the location of initial matrix crack initiation sites knowing the matrix failure stress.

Material	Temperature, ℃	Primary creep constant,* A	Primary creep stress exponent, n	Primary creep time exponent, p	Apparent activation energy, kJ/mol
CVI SiC/SiC (I)	1,315	9.05×10 ⁻⁵	1	0.33	619
CVI SiC/SiC (I)	1,450	9.05×10 ⁻⁵	1	0.33	619
CVI SiC/SiC (II)	1,450	1.66×10 ⁻⁴	1	0.33	619
CVI SiC/SiC (II)	1,482	1.66×10 ⁻⁴	1	0.33	619
CVI SiC/SiC (II)	1,550	8.77×10 ⁻⁴	1	0.33	619

TABLE V.-PRIMARY CREEP PARAMETERS OF CVI SiC/SiC (I) AND (II) COMPOSITES

*Stress is in MPa, time in hours



Figure 30.—Tensile creep behaviors based on global (applied) and local stresses (estimated from creep parameters): (a) CVI SiC/SiC (I) composites at 1,315 °C and (2) CVI SiC/SiC (II) composites tested at 1,450 °C. Downward arrows in the figures indicate failed specimens.

The typical creep behavior of PIP SiC/SiC composites shows three distinct regions: an initial elastic region, followed by a short primary creep region, and then an extended secondary region where the creep rate reaches a constant value. In addition, in these composites the primary creep region lasts for 1 to 20 hr at elevated temperatures and high stresses. Therefore, from a creep modeling point of view the primary creep can be neglected from Equation (6) and only secondary creep can be modeled. After deducting elastic and primary strains, Equation (6) can be reduced to the following relationship between the secondary creep strain (ϵ_{SC}), and time.

$$\varepsilon_{\rm SC} = B\sigma^{n'}t \tag{11}$$

where B is the secondary creep constant, n' is the secondary stress exponent, σ is applied stress, and t is the exposure time.

Differentiating Equation (11) with time yields,

$$\varepsilon^*{}_{\rm SC} = \mathbf{B}\boldsymbol{\sigma}^{\mathbf{n}'} \tag{12}$$

An Arrhenius-type rate model can then be used to account for temperature effects on secondary creep rate (minimum creep rate). Thus, equating secondary creep rate to rate equation,

$$\varepsilon^*_{\rm SC} = [D][e^{(-Q/RT)}] \tag{13}$$

where D is a constant and all other symbols are as defined earlier. Taking natural log on both sides of Equations (12) and (13), and plotting ln(creep rate) versus ln(stress) and ln(1/T), the secondary creep exponent (n') and the apparent activation energy (Q), respectively were determined. Knowing n' and Q, B was calculated at the tested temperatures. Table VI shows the measured secondary creep constants of PIP SiC/SiC composites at 1,315, 1,450 and 1,550 °C. A noticeable point from the table is that the secondary creep constant, B, increases with increase in temperature while all other creep constants remained relatively the same in the tested temperature range.

Even though creep of PIP SiC/SiC composites is controlled by the fibers, the difference between the average local and global applied stresses can still occur depending on microcrack density and pore size distribution in the PIP SiC matrix and the extant damage to the load bearing fibers during processing as well as during creep deformation. In addition, these microcracks can also grow during creep deformation depending on the stress and temperature. To determine the magnitude of stress enhancement occurring in these composites, an approach similar to that used for CVI SiC/SiC composites was adopted. Using creep parameters shown in Table VI, secondary creep behavior of PIP SiC/SiC composites at a fixed global (applied) stress was matched with the creep behavior predicted based on estimated local stress. Figure 31 shows secondary creep behaviors of PIP SiC/SiC composites at a fixed global stress end the corresponding predicted creep behaviors based on estimated local stresses (from the creep parameters). Two conclusions can be drawn from the figure: first, at a fixed stress, minimum creep rate vary significantly. Second, local stress in the composite can be 2 to 3 times greater than the global stress.

THELE VI. SECONDARY CREEK THRAMETERS OF THIS COMPOSITES				
Temperature, °C	Secondary creep constant,* B	Secondary creep stress exponent, n'	Apparent activation energy,	
			kJ/mol	
1,315	1.97×10^{-13}	4.01	953	
1,450	5.53×10 ⁻¹²	3.99	953	
1,550	2.23×10^{-10}	3.95	953	

TABLE VI.—SECONDARY CREEP PARAMETERS OF PIP SiC/SiC COMPOSITES

*Stress is in MPa, time in hours



Figure 31.—Secondary creep behaviors of PIP SiC/SiC composites at 1,450 °C based on global (applied) and estimated local stresses (predicted from the creep parameters)

4.0 Conclusions

In-plane tensile and creep properties of 2D woven, 0/90 balanced, CVI and PIP SiC/SiC composites with Sylramic[™]-iBN fibers were investigated at temperatures to 1,650 °C in air. Batch-to-batch variations in microstructure were characterized. Failure mechanisms under tensile and creep deformation modes have been identified. The creep parameters of both composites have been determined. The major findings are the following:

(1) Both composites exhibit wide variations in porosity and microstructure from batch-to-batch and within a batch, and do not show significant loss in in-plane tensile properties (PLS, E and UTS) up to 1,315 °C. Beyond this temperature, these properties decrease with increase in temperature. The average UTS values of both composites at a fixed temperature are nearly the same, although the volume fraction of fibers in the loading direction in PIP SiC/SiC composites (0.25) is greater than that in CVI SiC/SiC composites (0.18). This indicates that during processing, SylramicTM-iBN fibers in PIP SiC/SiC composites are degraded to a greater extent than those in CVI SiC/SiC composites.

(2) CVI SiC/SiC composites exhibit both matrix and fiber-dominated creep, and the creep mechanisms change depending on the applied stress and temperature. These composites do not show a minimum creep rate even after extended exposure. On the other hand, PIP SiC/SiC composites show fiber-dominated creep, and a single creep mechanism operates at different stresses and temperatures. These composites reach a steady-state (minimum) creep rate after a short exposure time. In both composites at a fixed temperature and stress, significant variations in creep behavior were observed. Variation in creep behavior is predominantly due to variation in porosity and stress raiser from region to region, both of which will cause the average local stress to be greater than the global applied stress. The creep parameters of CVI SiC/SiC composites fabricated by different vendors are different possibly due to different processing conditions used for CVI SiC infiltration, and minor compositional variation of CVI SiC. Accumulated creep strain has no significant effect on in-plane tensile properties of both composites as long as the SiC matrix or the SiC fibers are not damaged during creep deformation.

(3) Both LMP plot and the Monkman-Grant relationship can be used to predict creep durability of PIP SiC/SiC composites.

(4) Both CVI and PIP SiC/SiC composites do not show creep durability at high stresses. The maximum stress at which these composites can exhibit 500- and 100-hr durability at 1,450 and 1,550 °C, respectively, is 69 MPa.

(5) Creep behavior of both types of composites can be predicted based on their creep parameters, provided local stress effects are considered. Local stress elevation caused by microstructural inhomogeneities and stress raisers can be 2 to 3 times that of the global (applied) stress. When the local stress exceeds a critical value, the matrix may crack and cause crack growth and eventual failure of the composite due to oxidation of the interface coating and fiber, depending on oxygen ingress.

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