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Degradation of Continuous Fiber Ceramic Matrix Composites Under Constant-Load Conditions

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Abstract

Ten different ceramic matrix composite (CMC) materials were subjected to a constant load and temperature in an air environment. Tests conducted under these conditions are often referred to as stressed oxidation or creep rupture tests. The stressed oxidation tests were conducted at a temperature of 1454°C at stresses of 69 MPa, 172 MPa and 50% of each material's ultimate tensile strength. The ten materials included such CMCs as C/SiC, SiC/C, SiC/SiC, SiC/SiNC and C/C. The time to failure results of the stressed oxidation tests will be presented. Much of the discussion regarding material degradation under stressed oxidation conditions will focus on C/SiC composites. Thermogravimetric analysis of the oxidation of fully exposed carbon fiber (T300) and of C/SiC coupons will be presented as well as a model that predicts the oxidation patterns and kinetics of carbon fiber tows oxidizing in a nonreactive matrix.

Introduction

Ceramic matrix composites (CMCs), including carbon/carbon (C/C) composites, are promising materials for use in high temperature structural applications. This class of materials offers high strength to density ratios. Also, their higher temperature capability over conventional superalloys may allow for components that require little or no cooling. This benefit can lead to simpler component designs and weight savings. These materials can also contribute to increases in operating efficiency due to higher operating temperatures being achieved. The wide range of applications for CMCs includes combustor liners, turbomachinery, aircraft brakes, nozzles, and thrusters [*1-4*].

When evaluating CMCs for potential use in high temperature structural applications, the basic characterization of the material obtained from mechanical and environmental testing is important in understanding the fundamental properties of the material. These types of tests however may not be able to provide enough information on how the material will perform at its application conditions. This is especially true when the many application conditions may include variables such as high temperature, mechanical and thermal stresses, flowing gases, reactive environments, high chamber pressure, and material reactivity/recession (oxidation). The ultimate performance test is to insert a CMC component into its true application (i.e., in an engine). This

is often very expensive and impractical when dealing with developmental materials. Simpler, less expensive, and more practical test methods must be utilized. One type of performance test is stressed oxidation or creep rupture testing. This type of test allows materials to be screened at the stress, temperature and environmental conditions that it might see in a real application.

Stressed oxidation tests are proposed since stress may cause a material to behave very differently from when it is unstressed. Unstressed, high temperature, furnace exposure of a material in air may not give a true indication of how susceptible the material is to oxidation. Without sufficient thermal stresses, a mechanically unstressed material will have the same number (or absence) of cracks as it has in its as-received condition. In materials that are uncracked in their as-received condition, such as externally seal coated silicon carbide/silicon carbide (SiC/SiC), the interior will remain closed off from the outside oxidizing environment. The formation of a silica (silicon dioxide) scale on the surface will cover flaws and also protect the material from oxidation. Materials that have a high coefficient of thermal expansion mismatch between the reinforcing fibers and the matrix, such as carbon/silicon carbide (C/SiC), are thermally stressed upon cooling to room temperature from the processing temperature. These types of materials will be microcracked in their as-received condition. At exposure temperatures close to the processing temperature, thermal expansion and rapid silica formation can close the cracks and protect the carbon fibers and the pyro-carbon (pyro-C) interphase from oxidation. However, the material will still be prone to oxidation at lower temperatures where cracks remain open. When a constant load is added to the test conditions, the material may perform very differently. In materials, such as SiC/SiC, a stress above the first matrix cracking stress will induce cracks and make the material more prone to oxidation. In C/SiC materials, stresses will further open pre-existing cracks so that crack closing does not occur and the material is more prone to oxidation due to easier ingress of oxygen.

To obtain a better understanding of how a material might perform in certain application conditions, stressed oxidation (creep rupture) tests were conducted. In this paper, the results of stressed oxidation testing of ten different CMC materials are presented. Much of the analysis and discussion will then focus on the susceptibility of carbon fibers to oxidation and a finite difference model will be presented.

Experimental

In NASA Marshall Space Flight Center's CMC Material Robustness Program, stressed oxidation tests were conducted at NASA Glenn Research Center (GRC). Significant testing, which included tensile tests in air at 21, 1482, and 1648°C (70, 2700 and 3000°F), thermal expansion from -157 to1649°C (-250 to 3000°F) and oxidative fatigue, was conducted at Southern Research Institute (SRI). Only the results of GRC's stressed oxidation testing and SRI's tensile testing will be presented. Further information about all testing conducted on the ten materials can be found in a proceedings paper [*5*].

The constituents and processing parameters of the ten CMC materials that were tested are shown in Table I. The materials included C/SiC, C/C, SiC/SiC, SiC/SiNC, and SiC/C ceramic matrix composites. The material vendors were DuPont-Lanxide Composites (DLC, now AlliedSignal Composites Incorporated, ACI), B.F. Goodrich, Carbon-Carbon Advanced Technologies (CCAT), Dow Corning Corporation, Carbon Composites Inc. (CCI), and Synterials. All materials had a two-dimensional plain weave fiber architecture with the exception

of the Dow material, which had a 5-harness satin weave. All materials also had a pyro-C interphase except the Dow material, which utilized a proprietary interphase. The 15.2 cm. (6") length dog bone shaped coupons had cross-sectional areas in the gage region that were fairly consistent for a given material. However there was variation between the ten materials with the width ranging from 1.02-1.04 cm. (0.40"-0.41") and the thickness ranging from 0.25-0.43 cm. (0.10"-0.17").

Table I—*The material constituents and processing parameters of the 10 CMCs materials.*

	MATERIAL	MANU-FACTURER	YARN	YARN HT	FIBER ARCH.	YARN ENDS	INTERFACE COATING	MATRIX	DENSIF-ICATION	OXID. INHIB.	COATING
1	ENHANCED C-SiC	DUPONT LANXIDE	1K T-300	1700°C	2D PLAIN	19/IN	PyC ALPHA - 3	SiC	ISOTHERMAL CVI	YES	CVI protect.
2	STD C-SiC	DUPONT LANXIDE	1K T-300	NONE	2D PLAIN	19/IN	PyC ALPHA - 3	SiC	ISOTHERMAL CVI	NO	CVI protect
3	C-SiC	BFG	1K T-300	1800°C	2D PLAIN	19/IN	PyC	SiC	ISOTHERMAL CVI	YES	CVD SiC
4	CTD C-C	CCAT	1K T-300	1700°C	2D PLAIN	30/IN	PyC	C	PHEN. IMPREG	NO	PACK SiC
5	Hi NIC.-SiC	DUPONT LANXIDE	1K Hi NICALON	NONE	5 HS	17/IN	PyC ALPHA - 3	SiC	ISOTHERMAL	YES	NONE
6	Hi NIC-SiNC	DOW	0.5K Hi NICALON	NONE	2D PLAIN	16/IN	PROP. NON C	SiNC	PIP	"YES"	NONE
7	Hi NIC. - C	BFG	0.5K Hi NICALON	NONE	2D PLAIN	16/IN	PyC	C	ISOTHERMAL	NO	SiC
8	CTD C-C	CCAT / SYNTERIALS	1K T-300	1700°C	2D PLAIN	30/IN	PyC	C	PHEN. IMPREG	NO	Si_3N_4
9	CTD C-C	CCI	1K T-300	NONE	2D PLAIN	19/IN	PyC	C	PULSED CVI	NO	SiC
10	C-SiC w/cbs	DUPONT LANXIDE	1K T-300 1K T-300		2D PLAIN	19/IN	PyC	SiC	ISOTHERMAL CVI	YES	CVI protect.

Stressed oxidation tests of CMCs were conducted in an Instron 8500 Servo-Hydraulic load frame with hydraulic, water-cooled, wedge grips. Temperature was monitored through the use of thermocouples placed at the front face and rear face of the sample. Strain was monitored by extensometers with probes placed at both edges of the coupon within the gage section. A SiC susceptor placed circumferentially around the sample's gage region was used to heat the gage section of the tensile bar. Heat up time was typically 20 minutes. Once the test temperature of 1454°C (2650°F) was reached, the load was applied. The software allowed the load to be applied at an even rate such that the load is reached in one minute regardless of the size of the final load. Three samples of each material were tested at stresses of 69 MPa (10 ksi) and 172 MPa (25 ksi). Seven of the materials were tested at 50% of their ultimate tensile strength (UTS). Tests were conducted in an air environment. Coupons remained under constant load at elevated temperature until they failed or until 25 hours had passed. The samples that survived 25 hours were fractured at room temperature to determine their residual strength.

Thermogravimetric analysis (TGA) was also conducted to compare and contrast the susceptibility of carbon fibers (T300) to oxidation when in a matrix and when fully exposed to the environment. The TGA testing of the T300 carbon fiber consisted of cutting approximately 0.4 g from a spool of T300 that was bundled in a 1K tow (bundle of 1000 fibers). The fiber material was

then placed in an alumina basket and hung by a platinum wire in the TGA furnace. With the material in place, the furnace was brought to the desired temperature (ranging from 600°C to 1100°C). Tests were conducted in flowing oxygen at a rate of 100 cc/min. flowing from a 1" diameter tube. Weight loss and time were recorded. The tests were conducted until all carbon was consumed. TGA tests were also conducted on a C/SiC composite material, specifically DuPont-Lanxide Composites' plain woven standard C/SiC material. The material had a chemically vapor deposited pyro-carbon interphase on the fibers and a chemically vapor infiltrated SiC matrix. A final chemically vapor deposited external seal coating of SiC was applied to the outside of the machined test coupons. The test coupons had dimensions of about 2.54 cm. x 1.27 cm. x 0.32 cm. (1.00" x 0.50" x 0.12") and had weights of approximately 2.4 g. The amount of the carbon fiber within the composite was about 42 wt. %. At the desired temperature (ranging from 550°C to 1400°C), TGA tests were conducted in oxygen at the same flow rate as for the fully exposed T300 fiber. The TGA tests of the C/SiC coupons were conducted for a duration of 25 hours. The results of TGA tests will be presented in the discussion.

Stressed Oxidation Results

In order to interpret better the results of stressed oxidation testing, the ultimate tensile strength of the materials should be known. The results of tensile tests conducted by SRI at 21, 1482, and 1648°C (70, 2700 and 3000°F) are shown in Figure 1. As illustrated by the strengths at elevated temperatures, the C/SiC composites typically have higher strengths than the SiC/SiC composites. The specific C/C materials supplied by the venders show some variation in strength.

The times to failure for the samples stressed oxidation tested at 69 MPa are shown in Figure 2. Three of the materials survived 25 hours of exposure. The three materials (DLC enhanced C/SiC, DLC C/SiC w/cbs, and Dow SiC/SiNC) all had oxidation protection enhancements which, seem to be a key in prolonging the life of both C/SiC and SiC/SiC composites. The DLC enhanced C/SiC material has a boron containing particulate in the composite that acts as an oxygen getter and forms borosilicate glasses that seal cracks and protect the fibers from oxidation. The DLC C/SiC material with a cbs (carbon, boron, and silicon) coating uses a similar method of oxidation protection as the enhanced C/SiC material, however, a boron containing coating also provides an exterior form of protection. The Dow SiC/SiNC uses a proprietary, non-carbon, interphase on the SiC fibers. The three materials had relatively high residual strengths as determined from room temperature tensile tests. Two other materials, CCAT C/C and DLC SiC/SiC, did fail within 25 hr, but had relatively longer lives compared to the five other materials that failed at shorter times.

The times to failure for testing at a stress of 172 MPa are shown in Figure 3. All of the materials either failed on load-up or had much shorter lives than at 69 MPa with the exception of the DLC C/SiC material with the cbs coating. This material had lives over ten hours while the other materials either failed load-up or had lives of a few minutes or about half an hour. The cbs coating enhancement in the DLC material seems to be effective in sealing cracks and protecting the fibers even at stresses as high as 172 MPa. It should be noted that the DLC enhanced C/SiC and the DLC C/SiC w/cbs materials had failures in a thermal gradient region located in between the heated gage section and the water-cooled grips. This suggests that the enhancements in these materials are effective at our specific temperature of 1454°C, however the materials may be susceptible to degradation at intermediate temperatures.

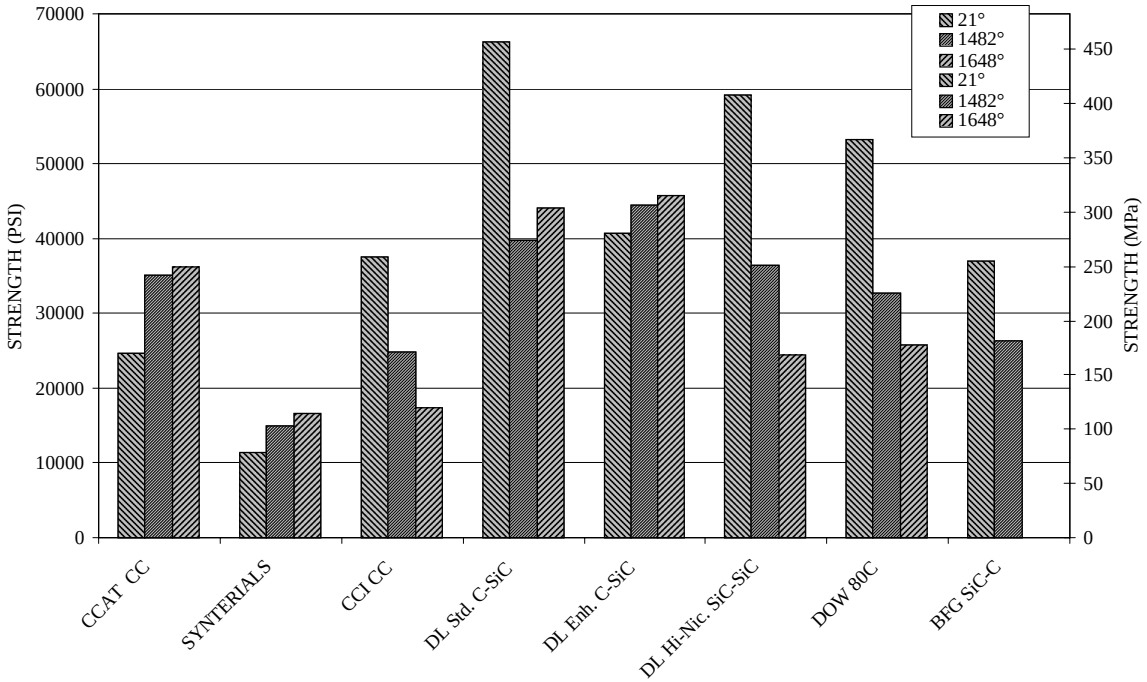

Figure 1—*The tensile strength of eight of the program materials at temperatures of 21, 1482 and 1648°C (70, 2700 and 3000°F). Tensile testing conducted by SRI.*

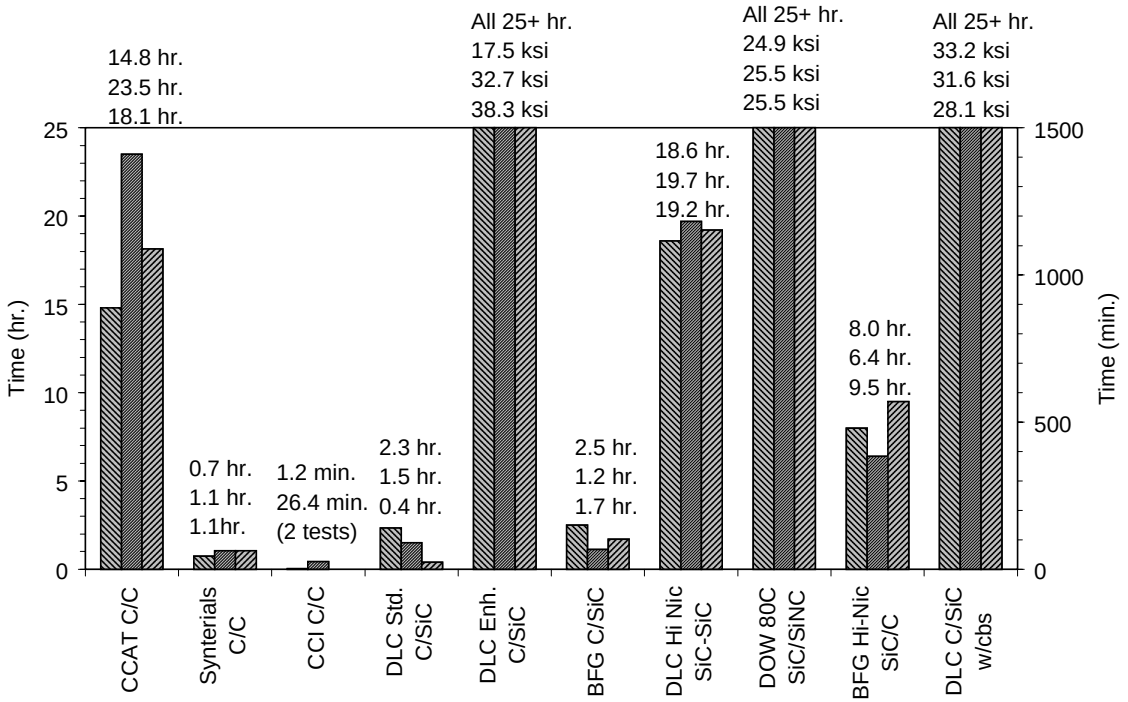

Figure 2—*Times to failure for stressed oxidation tests at 1454°C/69 MPa.*

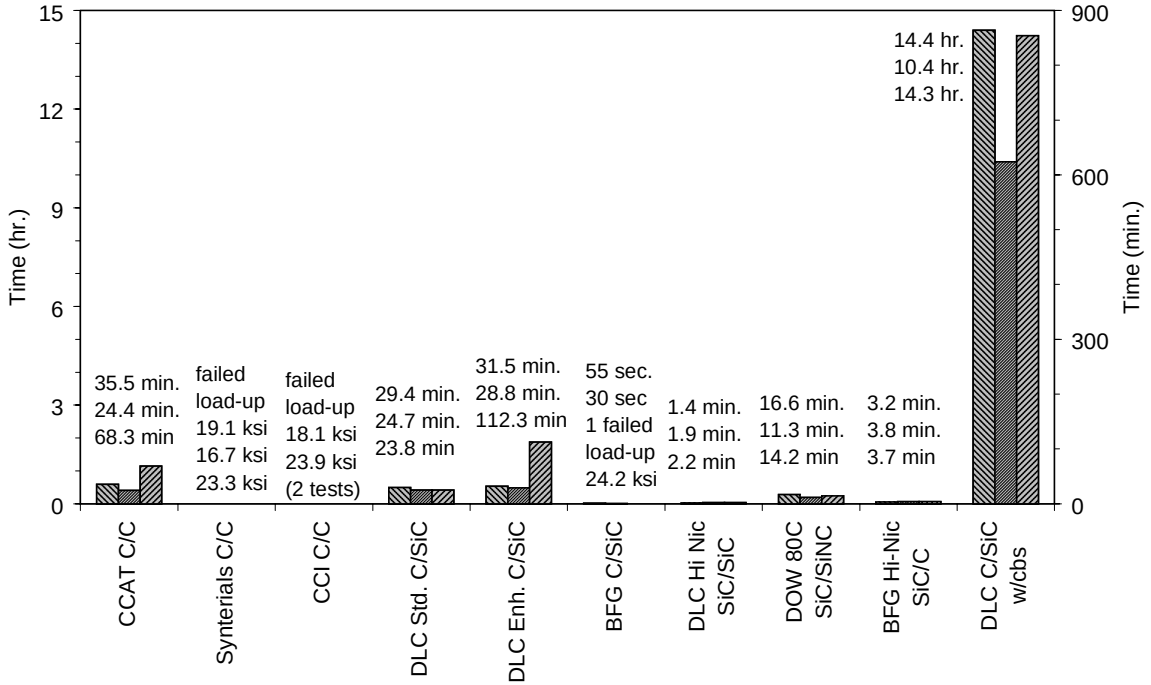

Figure 3—*Times to failure for stressed oxidation tests at 1454°C/172 MPa.*

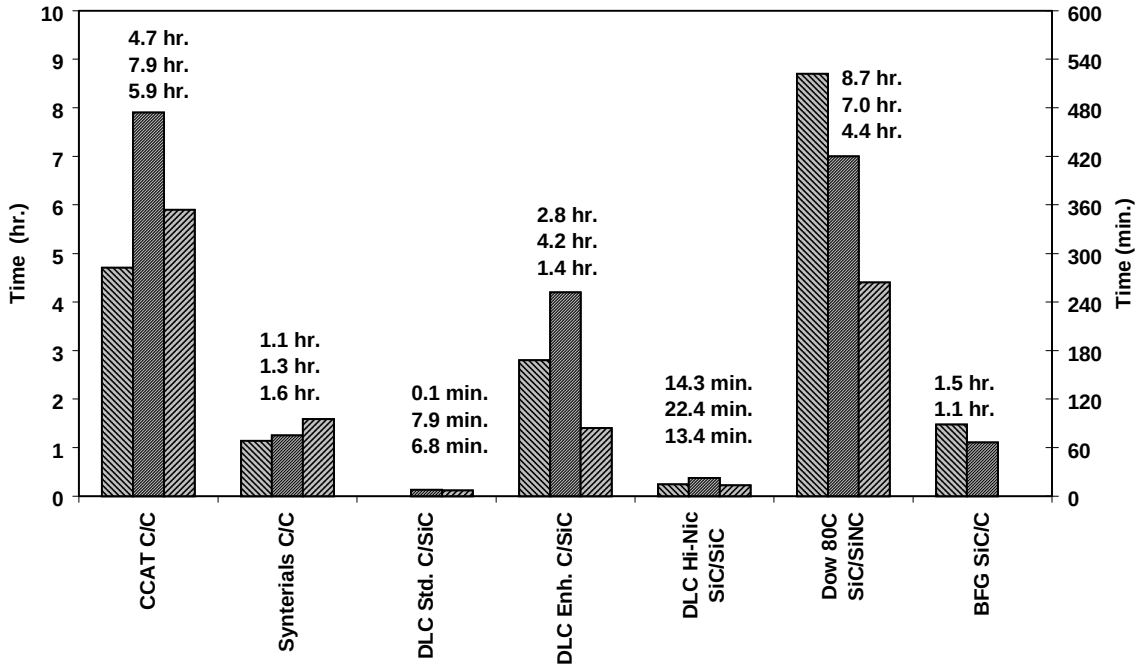

Figure 4—*Times to failure for stressed oxidation tests at 1454°C/50% UTS.*

Stressed oxidation was also conducted at stresses of 50% of the ultimate tensile strength of each material. The times to failure are shown in Figure 4. Testing at this stress was conducted so that all materials could be evaluated at a stress that seemed to be fairer for all materials regardless of the maturity of the material or the material type. However 50% of the UTS is likely above the first matrix cracking stress for the SiC/SiC and SiC/SiNC materials. Of the seven materials tested at this stress, two materials did better than the rest (CCAT C/C and Dow SiC/SiNC).

The conditions that the materials are exposed to under stressed oxidation tests are harsh enough that the susceptibility of the material to oxidation, creep, and mechanical damage can be determined. Post-test material characterization can help determine the extent and source of damage and which constituents within the CMC are more susceptible. Microstructural analysis of fracture surfaces using a scanning electron microscope and of polished cross-sections using an optical microscope is underway and will be reported in the near future. The remainder of this paper will focus on C/SiC composites and their behavior while under constant load conditions.

TGA Tests

Before the effect of a constant load on C/SiC composites is evaluated, it is beneficial to have an understanding of how the material and its fiber constituent behave in unstressed, high temperature, oxidizing conditions. The percent weigh loss of T300 carbon fiber versus time at temperatures ranging from 600-1100°C is shown in Figure 5. Slower reaction rates are seen for temperatures ranging from 600-750°C. Faster reaction rates and less of a temperature dependence are observed at temperatures above 800°C. At all temperatures, the approximately 0.4 grams of carbon fiber had completely oxidized away within an hour. These same trends were observed in thermogravimetric analysis (TGA) experiments on T300 fiber conducted by Lamouroux et al [*6*].

The oxidation of the fully exposed T300 fiber is contrasted by the TGA results of externally-seal coated C/SiC test coupons as shown in Figure 6. At low temperatures, oxygen is able to diffuse into the pre-existing cracks within the composite and react with the carbon fibers. At 550°C, the start of a downward trend in weight loss is observed however the test had to be stopped early. At 750°C, oxidation occurs sooner and at a faster rate. Complete fiber burnout was observed to occur within 5 hours. However, at the higher temperatures, the trends in weight loss and complete fiber burnout did not continue as observed in the TGA tests conducted on the bare T300 fiber. As test temperatures were increased, weight loss rates greatly decreased so that low weight losses were observed even after 25 hours. It should be noted that two tests were conducted at 1000°C and both had different oxidation rates, which are believed to be due to the difference in pre-existing cracks in the external seal coating. Microstructural analysis of polished cross-sections revealed that one sample oxidized uniformly from the outer edges toward the interior while in the other sample significant oxidation occurred from one edge toward the interior but at the opposite edge little oxidation occurred.

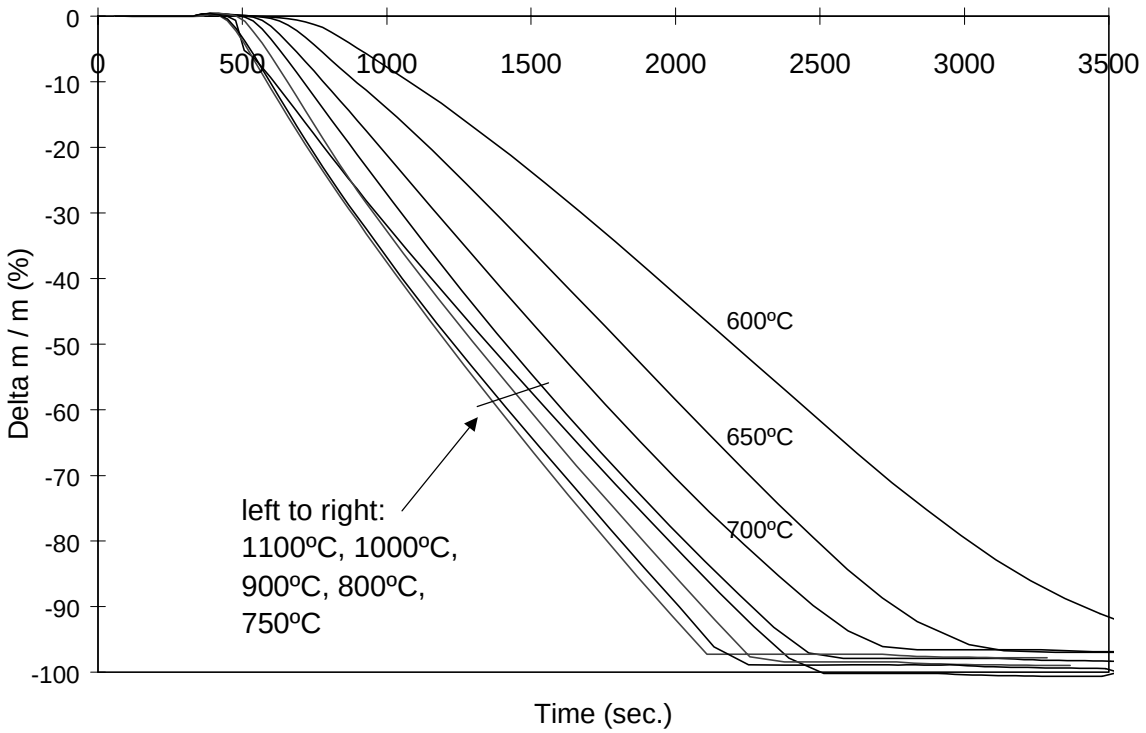

Figure 5—*Weight loss versus time of T300 carbon fiber in flowing oxygen at temperatures ranging from 600–1100°C.*

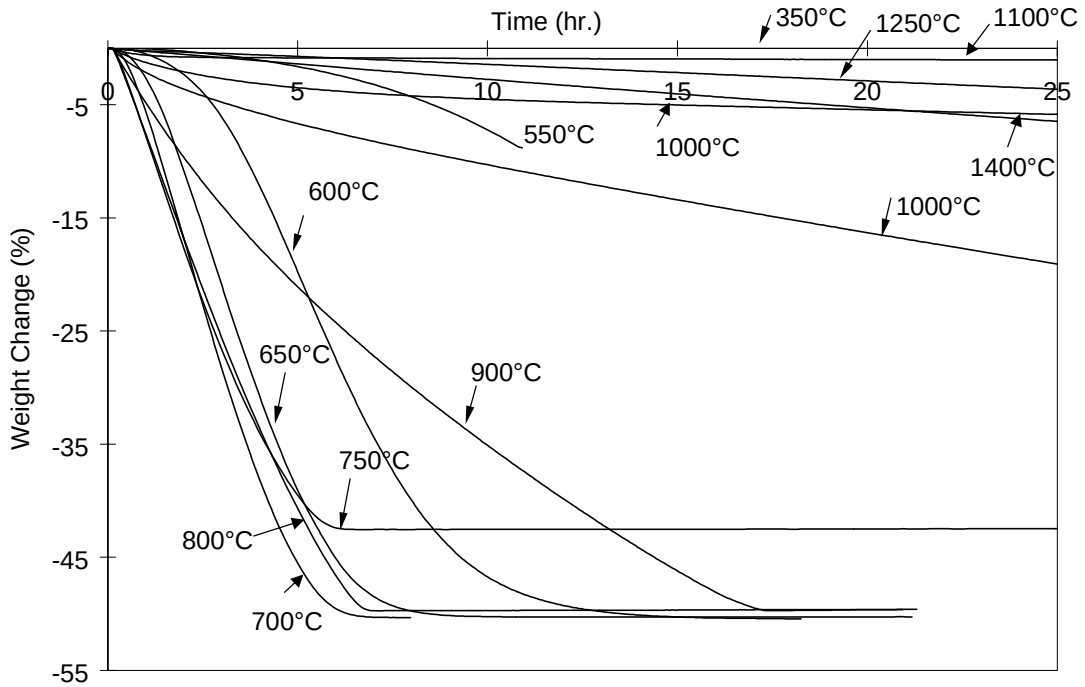

Figure 6—*Weight loss versus time of standard C/SiC in flowing oxygen at temperatures ranging from 550–1400°C.*

The protection of interior fibers due to the formation of silica and crack closure has been observed by other researchers [*7-9*]. This effect is due to two factors, which aid in stopping or significantly decreasing the supply of oxygen into the interior of the composite. The first factor is due to crack edges coming very closely together at temperatures at or near the processing temperature (typical CVI processing performed around 1100-1250°C). As the fiber and matrix expand to their original lengths at the processing temperature, residual thermal stresses are relieved and the pre-existing microcracks become narrower and pinch off the supply of oxygen to the fibers. The second factor is due to the formation of silica as oxygen reacts with silicon carbide. The oxide is in the form of a scale and can cover the surface of the composite and also fill in microcracks. The growth of the oxide scale can act as a sealant at high temperatures. In an unstressed condition, carbon fibers within a C/SiC CMC are therefore more susceptible to oxidation at lower temperatures than at the higher temperatures where thermal expansion, oxide scale growth, and enhancements are effective in closing cracks and cutting off the supply of oxygen to the fibers. However, some oxidation is still to be expected at high temperatures due to the slow growth of oxides compared to the rate at which carbon oxidizes. TGA experiments on chemically vapor deposited SiC at temperatures of 1200-1500°C and at one atmosphere in air were conducted by Ogbuji and Opila to study the oxidation kinetics of SiC. They determined the thickness of the oxide scale on SiC after oxidation at 1300°C for 75 hr to be 1.6 micrometers [*10*]. In addition to the slow rate at which silica scales grow, enhancements may also be slow in effectively sealing cracks.

Constant Load

When a constant load is applied to C/SiC at elevated oxidizing temperatures, the applied stress opens existing cracks and allows for easier ingress of oxygen to the fibers. At sufficiently high stresses, cracks may be open too wide for crack closure and sealing to occur. The results of stressed oxidation of DLC's standard C/SiC material at stresses of 69 MPa and 172 MPa and at temperatures ranging from 750°C-1500°C are shown in Figure 7 [*11*]. This batch of material is two years older than the DLC standard C/SiC material tested under the NASA MSFC CMC Material Robustness Program.

In the strain versus time curves, the effect of stress can be seen. As expected, the samples tested at the higher stress had shorter lives. The samples tested at 172 MPa had curves that fall to the left side of the plot while the samples tested at 69 MPa had longer lives and were concentrated to the right side of the plot. Although the times to failure across the temperature range occurred within a fairly narrow range, times to failure increased from 750°C to 1250°C and then decreased at temperatures higher than 1250°C for both stresses. Another effect from temperature is the strain to failure. Samples tested in the higher temperature range showed a much more pronounced tertiary regime and higher strains to failure.

Carbon has been observed to oxidize at temperatures as low as 500-550°C [*6,12,13*]. Stressed oxidation of C/SiC at 550°C did show strength degradation due to oxidation. A sample tested at 172 MPa failed in 25 hours while a sample tested at 69 MPa survived 25 hours. The residual strength of the surviving sample was determined at room temperature to be only half that of the as-received material. The strength reduction and failure at 550°C was not attributed to mechanical damage from the sample being held at a constant load. This conclusion is based upon similar tests conducted in air at 350°C and in argon at 750°C. During 25 hours at load (69 and

172 MPa) and temperature, no significant change in strain due to creep was observed. After 25 hours of exposure, the tests were stopped. The residual strengths determined at room temperature were compatible with the tensile strength of the as-received material [*11*].

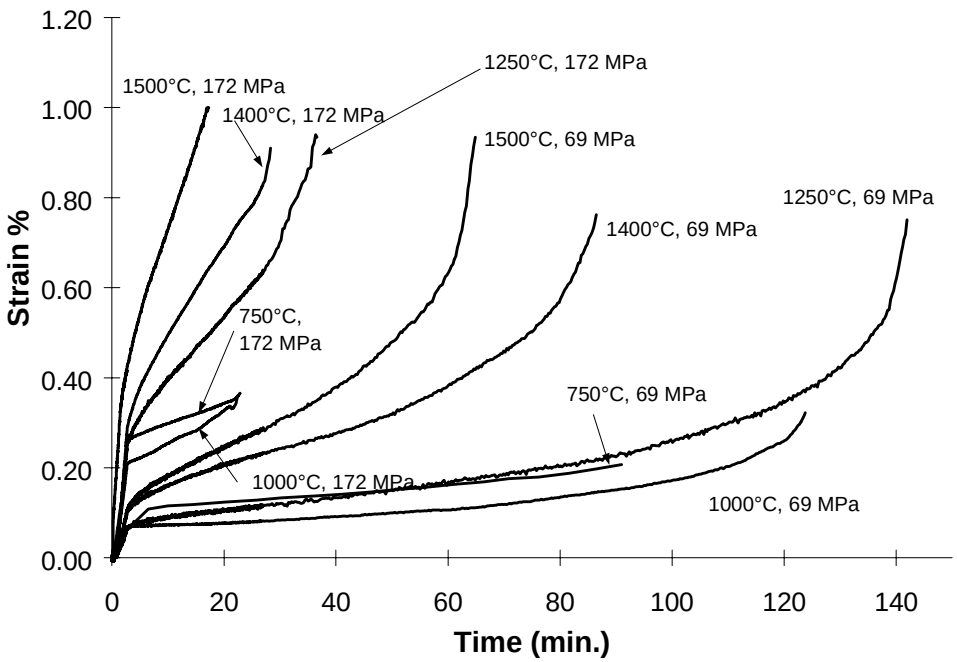

Figure 7—*Strain versus time curves from stressed oxidation testing of C/SiC at stresses of 69 MPa (10 ksi) and 172 MPa (25 ksi) and temperatures from 750–1500°C.*

Modeling

Microstructural analysis of oxidation patterns in tested samples, times to failure of C/SiC in stressed oxidation testing, and reaction rates of T300 carbon fiber in TGA experiments suggests two types of kinetics, reaction controlled and diffusion controlled. In order to gain a better understanding of the oxidation of carbon within a CMC and the controlling kinetics, a finite difference model has been developed.

Only the results from the model and a comparison to experimental observations will be presented. Additional information about the model can be found in proceedings papers [*14,15*]. The model assumes the case of an 8 x 8 array of carbon fiber tows that bridge a cracked matrix. The 2-D model takes the case of pre-cracked as-received C/SiC and simplifies the many microcracks within the material by representing them as one continuous crack that spans the entire surface so that fibers bridge the open plane of the cracked matrix. The model is run at a given Sherwood number.

The Sherwood number is a dimensionless parameter that is equal to the reaction rate constant (K [m/s]) times a distance (Δx [m]) divided by the diffusion coefficient (D [m^2/s]). The reaction rate constant and the diffusion coefficient both have a temperature dependence. Across the temperature range of 550-1500°C, theoretical calculations show that the diffusion coefficient will change by a factor of three while the reaction rate constant changes by three orders of magnitude. The calculations for the reaction rate are based on a carbon activation energy of 100 kJ/mol and use linear recession rate data from Eckel et al [*16*] to solve the Arrhenius

reaction rate equation. The diffusion coefficients were calculated using Chapman-Enskog kinetic theory. Because of the temperature dependencies, the ratio of the value for the reaction rate constant to the diffusion coefficient will give high or low Sherwood values. At low temperatures, relatively low Sherwood numbers are obtained since the value of the reaction rate constant will be significantly lower and the value for the diffusion coefficient will only be slightly lower than their mean values in the temperature range of 550–1500°C. Therefore at low temperatures, the kinetics will be controlled by the lower or more limiting step of the rate of carbon-oxygen reactions, i.e. reaction controlled. At high temperatures, the reaction rate will be significantly higher and the diffusion coefficient will only be slightly higher than the mean values within the temperature range. This would give relatively high Sherwood numbers. In this case, the kinetics will be dependent on the rate at which oxygen is supplied to the carbon, i.e. diffusion controlled kinetics. For intermediate temperatures, mixed control kinetics may be observed as the kinetics are in transition from reaction control to diffusion control or vice versa.

In the model, a 170 x 170 grid pattern is laid out with an 8 x 8 fiber tow array in the middle of the matrix. The tows are ten grids in diameter and are spaced 10 grids apart. Therefore, the grids within the 170 x 170 grid pattern will either be open grids within the fiber bridged region where gas phase diffusion can occur or the grids will represent cross-sections of fibers that bridge the crack. The outer edge is maintained at a constant oxygen concentration due to atmospheric conditions. Oxygen is able to diffuse into the matrix until a quasi-steady-state is reached. The local oxygen concentration of each open grid is determined by sampling from its four neighboring grids over hundreds of iterations until steady state is reached. In a method used by Glime and Cawley [*17*] to study profiles of oxidized carbon fiber tips, carbon is removed incrementally. The smallest time increment needed to oxidize away the carbon from a grid is determined across the whole array and oxidation is then allowed to occur for that amount of time. Then the whole process of oxygen diffusion until quasi-steady state is reached, determining the smallest time increment, and oxidation for that amount of time is repeated over several iterations until the desired amount of carbon has been reacted away.

The model was run at values (i.e. reaction rate constant, diffusion coefficient, and total oxygen concentration at the edge) correlating to conditions at 1400°C in air, and was compared to the microstructure of a tested coupon. Figure 8 shows the polished cross-section of a sample that had been stressed oxidation tested at 1400°C/69MPa. Severe carbon consumption is seen around the perimeter of the sample (darkened regions in the micrograph). In the close-up views, a reaction front can be seen as carbon is immediately oxidized as soon as oxygen is supplied so that the interior is starved in oxygen and no oxygen is present to react with carbon in the interior of the matrix. In the close-up view of the interior, a large void and cracks in the fiber tows are seen, however, no evidence of oxidation is observed. The oxidation patterns shown in Figure 8 suggest a steep gradient in oxygen concentration at the reaction front toward the interior of the sample. This is an oxidation pattern that would be expected from diffusion controlled kinetics. The results from running the model at conditions for 1400°C are shown in Figure 9. The oxidation pattern on a 2-dimensional surface within the fiber bridged region is shown at various stages in the top row of plots. A shrinking core effect is seen as fibers on the outer perimeter are consumed first and then the reaction front moves inward to the next perimeter of fibers. The interior fiber tows remain unreacted until the oxygen supply reaches them. The correlating oxygen concentration plots for one-quarter of the 2-dimensional section are shown in the bottom row. A steep gradient in oxygen concentration can be seen at the edge of the reaction front

toward the interior of the sample. The interior is shown to be deprived of oxygen. These patterns agree well with experimental observations and suggest diffusion controlled kinetics at this relatively high temperature.

The model was also run at values correlating to conditions at 700°C, and were compared to the polished cross-section of a test coupon that had been stressed oxidation tested at 750°C. Figure 10 shows the polished cross-section of a sample stressed oxidation tested at 750°C/69 MPa. Minimal and uniform oxidation is seen along the edge of the sample as well as deep in the interior of the sample along the edges of fiber tows (bundles) and along the matrix cracks within the tows. This pattern suggests that the carbon/oxygen reactions are so slow that oxygen is able to bypass the carbon at the outer edge and saturate the interior at high concentrations so that oxygen is available throughout the matrix to supply the slow reaction process. This is the oxidation pattern that would be expected for reaction controlled kinetics. The results from running the model at conditions for 700°C are shown in Figure 11. The oxidation pattern of the bridging fiber tow array on the 2-dimensional surface is shown in various stages in the top row of plots. The correlating oxygen concentration plots for one-quarter of the section are shown in the bottom row. Uniform carbon reactions and high oxygen concentration are seen throughout the 2-dimensional surface of the matrix. These patterns agree well with experimental observations and suggest reaction controlled kinetics at this relatively low temperature.

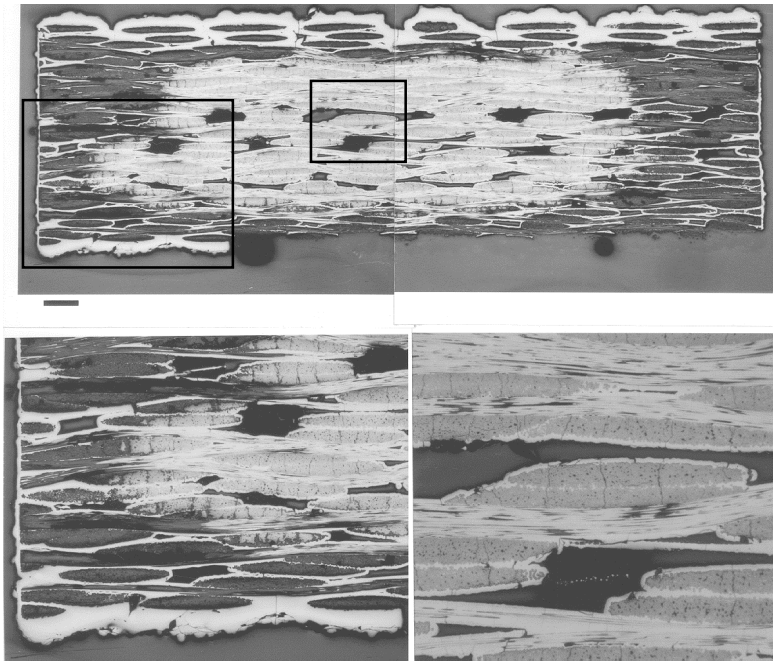

Figure 8—*Diffusion controlled kinetics. Optical micrograph of a polished cross-section of a C/SiC sample stressed oxidation tested at 1400°C/69 MPa. Time to failure was 86 minutes. Scale bar in top micrograph equals 0.5 mm.*

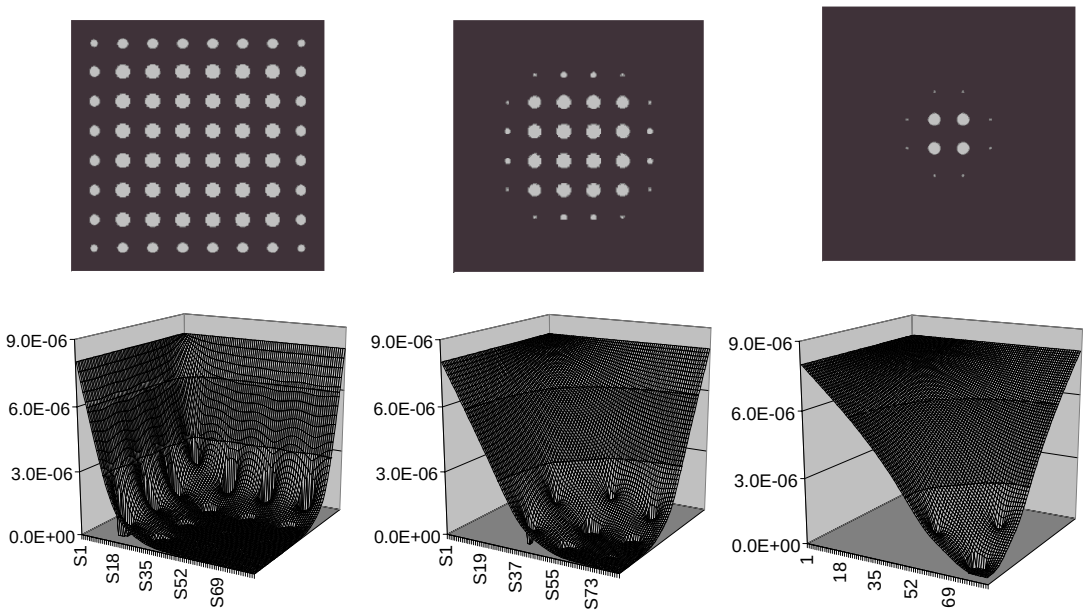

Figure 9—*Diffusion controlled kinetics for Sh= 1.07 (relating to 1400°C) at 25%, 75%, and 95% carbon consumption of the 8 x 8 fiber array. The top illustrations are of the remaining area of carbon on the 2-dimensional surface. The bottom plots are of the related oxygen concentrations (mol//cm3) for one quarter of the surface.*

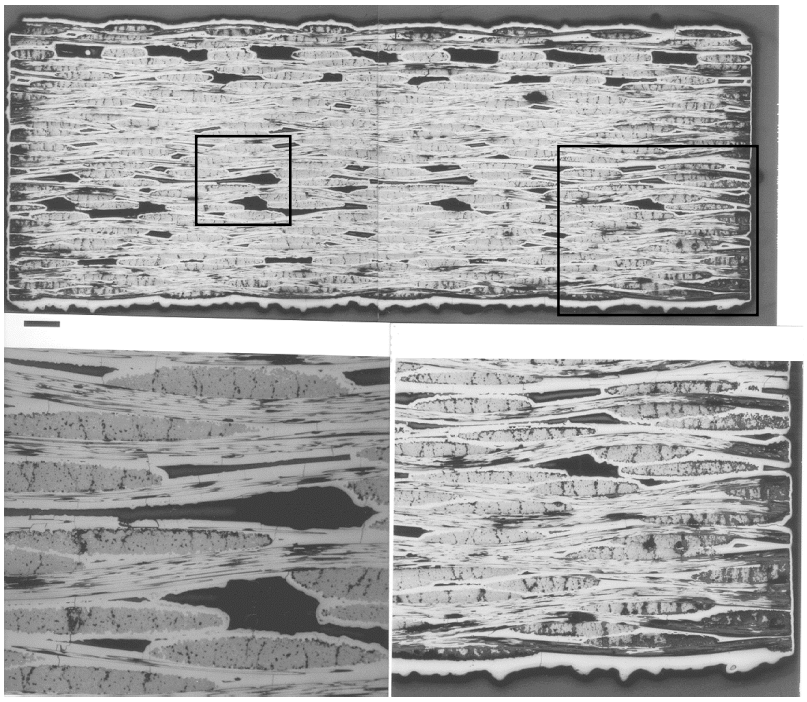

Figure 10—*Reaction controlled kinetics. Optical micrograph of a polished cross-section of a C/SiC sample stressed oxidation tested at 700°C/69 MPa. Time to failure was 91 minutes. Scale bar in top micrograph equals 0.5 mm.*

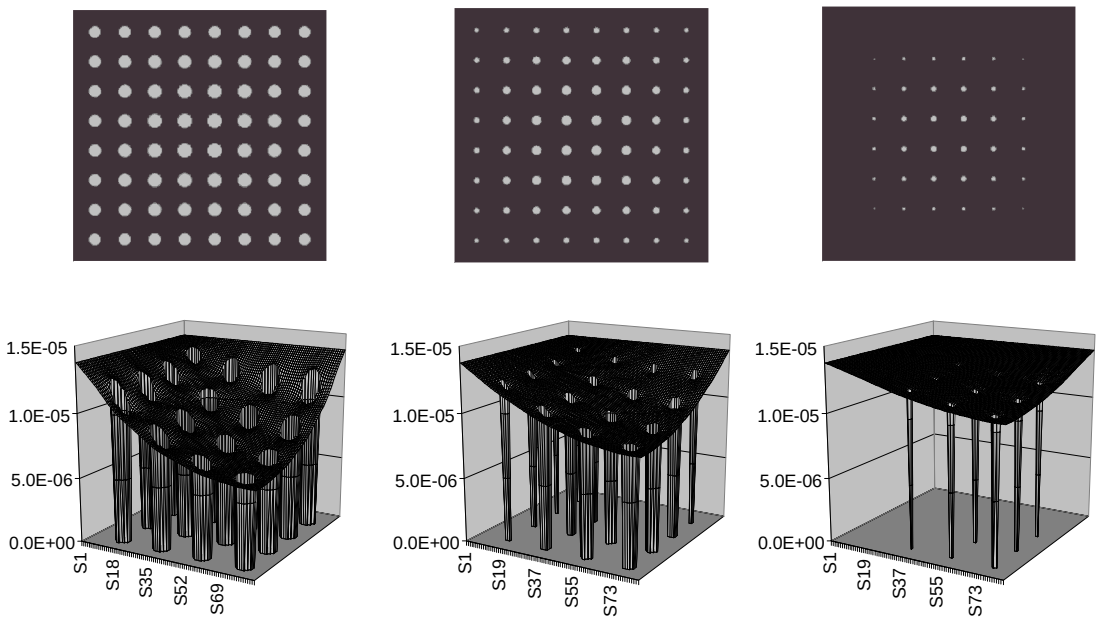

Figure 11—*Reaction controlled kinetics for Sh=1.57E-2 (relating to 700°C) at 25%, 75%, and 95% carbon consumption of the 6 x 6 fiber array. The top illustrations are of the remaining area of carbon on the 2-dimensional surface. The bottom plots are of the related oxygen concentrations (mol/cm^3) for one quarter of the surface.*

Conclusions

CMCs can behave very differently in stressed and unstressed conditions in air at elevated temperatures. Unstressed materials may oxidize considerably less than when a stress is applied. Cracks may not be present or may be closed when the materials are unstressed. However when stressed, materials can be greatly damaged due to mechanical stresses, oxidation, and creep. It was observed that the addition of oxidation inhibitors or enhancements can significantly increase the performance of a material. Stressed oxidation and microstructural analysis of C/SiC samples and TGA tests of T300 fiber show different trends in oxidation patterns, times to failure and fiber reactivity at temperatures above 800°C compared to the results obtained at temperatures below 800°C. These trends suggested reaction controlled kinetics at low temperatures and diffusion controlled kinetics at high temperatures. The finite difference model compared well with experimental tests and microstructural analysis in predicting oxidation patterns and kinetics.

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13. ABSTRACT ***(Maximum 200 words)***
Ten different ceramic matrix composite (CMC) materials were subjected to a constant load and temperature in an air environment. Tests conducted under these conditions are often referred to as stressed oxidation or creep rupture tests. The stressed oxidation tests were conducted at a temperature of 1454 °C at stresses of 69 MPa, 172 MPa and 50% of each material's ultimate tensile strength. The ten materials included such CMCs as C/SiC, SiC/C, SiC/SiC, SiC/SiNC and C/C. The time to failure results of the stressed oxidation tests will be presented. Much of the discussion regarding material degradation under stressed oxidation conditions will focus on C/SiC composites. Thermogravimetric analysis of the oxidation of fully exposed carbon fiber (T300) and of C/SiC coupons will be presented as well as a model that predicts the oxidation patterns and kinetics of carbon fiber tows oxidizing in a nonreactive matrix.

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