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MECHANICAL TESTING OF A COATED CARBON/CARBON COMPOSITE AT ELEVATED TEMPERATURES IN AIR

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CERAMICS RESEARCH DIVISION

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ABSTRACT

The strength of a carbon/carbon composite having a proprietary silicon carbide coating with silicate glass interlayers was evaluated at ambient and elevated temperatures in air. The coating was applied by the controlled nucleation thermochemical deposition (CNTD) process. The strength, static fatigue, and oxidation results of the coated composite were favorable at temperatures up to 2372°F (1300°C) in air.

FOREWORD

This report is in accordance with draft guidelines for the release of Composites Technology issued by the Associated Deputy Under Secretary of Defense (Technology Transfer). This report contains data on basic material behavior and properties, not revealing know-how related to production or end item application and is accordingly releasable in open exchange.

CONTENTS

INTRODUCTION

The controlled nucleation thermochemical deposition (CNTD) process[1-3] can apply a silicon carbide coating to carbon/carbon (C/C) composites. Coated C/C composite materials are potentially useful in advanced gas turbine engines, particularly as static components, although rotating components are feasible.[4] Success of such a system will require a coating that will ensure oxidation resistance and mechanical integrity. This report represents a preliminary characterization of mechanical properties of a silicon carbide coated, C/C composite at ambient and elevated temperatures in air.

Another study of coated C/C composites is summarized in Reference 4. It was noted that systems with silicon carbide barrier alone offered only short duration (5 to 10 hours) protection, particularly in the dynamic (2500°F) hot gas environment. Alternatively, systems with a silicon carbide barrier in conjunction with a silicate glass forming barrier layer (to provide flaw-healing capability) were the most durable.[4]

The present study evaluates such a composite system with a multilayer coating. The material is similar in concept to and a subsequent development to the Aerojet-Bridge material described in Reference 4. Mechanical test evaluation is at elevated temperature in air as well as at room temperature.

MATERIALS

The C/C composite grade T-300* was obtained in the form of a 0.25" x 5" x 14" plate. T-300 is a two-dimensional laminate with continuous carbon fiber plies arranged in a 0 to 90° weave. The plate was cut into 145 flexure bar substrates having nominal dimensions of 0.110" x 0.150" x 3" or 3.5". Most bars were given a duplex coating system, which contained an initial layer (interlayer) and a top or overcoat of ultrafine grained SiC (grade CM400).† The latter is a strong, oxidation and erosion-resistant composite of Si and SiC (β form) having a grain size of hundreds of angstroms. The interlayer's functions are not well understood, but it generally improves the adhesion of the SiC layer and provides a source of crack filling/healing material which contributes to oxidation resistance at temperatures lower than the deposition temperature of the top coating (typically 1000°C to 1200°C). Details of the coating system are proprietary, but the interlayer and overcoated are applied in two separate deposition runs, the final SiC layer at approximately 1200°C. Processing time at this temperature is approximately two hours for both the interlayer and the overcoat. The interlayer is a boron-rich glass forming material which is intended to provide a flaw-healing

*Hitco Corporation, Gardena, California.
†San Fernand Laboratories, Pacomia, California.

1. HOLZL, R. *Grain Refinement by Thermochemical Means* in Proceedings of the Sixth International Conference on Chemical Vapor Deposition, L. Donaghey, P. Rai-Choudhury, and R. Tauber, ed., The Electrochemical Soc., Princeton, NJ, 1977, p. 107-114.
2. DUTTA, S., RICE, R. GRAHAM, H., and MENDIRATTA, M. *Characterization and Properties of Controlled Nucleation Thermochemical Deposited (CNTD) Silicon Carbide.* NASA Technical Memorandum 79277. Presented at 80th Annual Meeting of the American Ceramic Society, Detroit, MI, 6-11 May 1978.
3. STIGLICH, J., BHAT, D., and HOLZL, R. *High Temperature Structural Ceramic Materials Manufactured by the CNTD Process.* Ceramurgia International, v. 6, no. 1, 1980, p. 3-10.
4. KEISER, R. *Oxidation Protection for High Strength Carbon/Carbon Composites.* Air Force Wright Aeronautical Laboratory, AFWAL TR-82-4060, June 1982.

capability. The two materials could be deposited consecutively in the same deposition run without changing chambers, but more efficient scheduling of staff and equipment results in a two-run procedure. The bars were heated by radiation within a furnace chamber incorporating a standard chemical vapor deposition setup.

The coating system was applied to two groups of 50 specimens at a time using SiC pins to hold them during deposition. Another 30 specimens were coated all over without using the pins, which are inconvenient and have been observed to lead to failure during oxidation testing. These had a 1/8"-diameter hole in the substrate end, through which a wire was used to hold the specimen in the deposition apparatus. The specimen-to-specimen coating thickness (interlayer plus overcoat) varied between 0.005" and 0.014". The variation in the thickness of the coating on an individual specimen was 0.002" to 0.004". The coating cross-section dimensions were measured on all specimens to estimate the coating stresses. The typical shape, dimensions, and appearances of the specimens are shown in Figure 1. The roughness of the substrate is generally replicated by a CNTD deposit unless a concerted effort is made for elimination it. Much work on substrate surface grinding would have been needed to improve the coating surface morphology. Figure 2 shows the SiC layer had cracks and was not continuous.

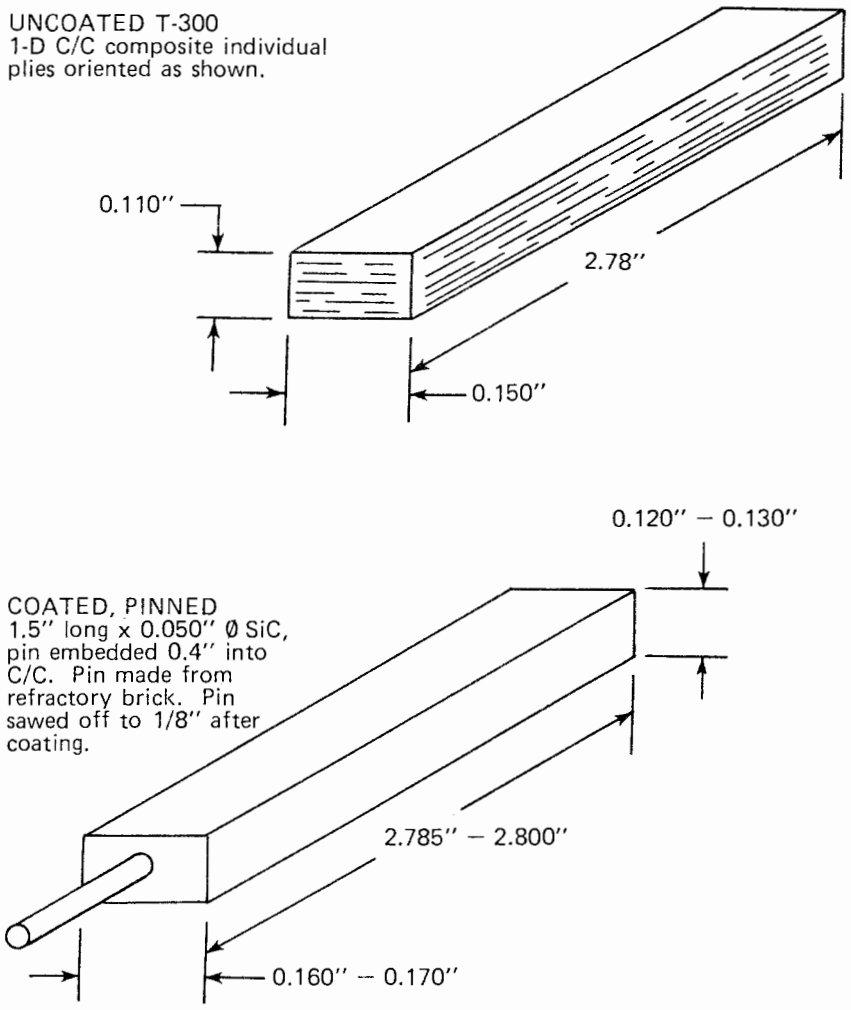
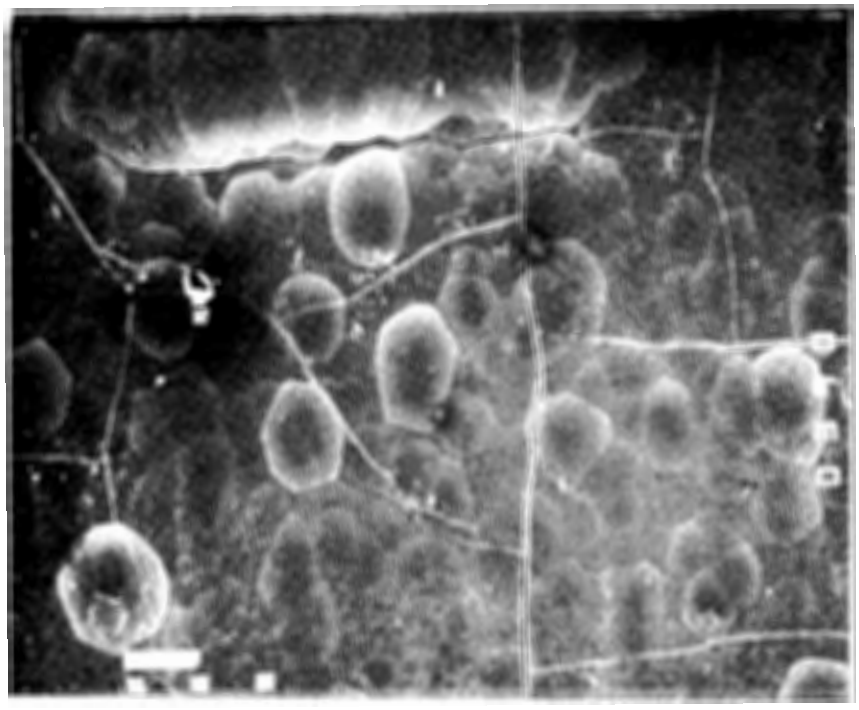


Figure 1. Uncoated and coated flexure bar specimens, typical dimensions.

a. Viewing direction ∥ to cloth plies, bar = 1000 μm



b. Viewing direction ⊥ to cloth plies, bar = 100 μm

Figure 2. SEM photos of the surfaces of a coated bar.

MECHANICAL TESTING PROCEDURES

Experiments performed included the following: short-time room temperature and elevated temperature four-point flexure tests in air; elevated temperature stress rupture and stepped temperature stress-rupture tests in air using four-point flexure; cyclic thermal shock (100 cycles) from 1200°C to room temperature (in an air jet) followed by room temperature flexure testing as above. All tests and apparatus have been described previously.[5-8]

Most flexural testing was carried out with C/C plies perpendicular to the applied external forces; this is the preferred mode of application in a real component. A few bars were tested in the fast fracture flexure tests with the C/C plies parallel to the applied forces. Maximum loads (and coating stresses) were much higher (1.5X typically) than for the former orientation. The maximum stress (σ) borne by the coating was approximated by Equation 1:

$$\sigma = \frac{3Pah_1}{b_1h_1^3 - (1-n)b_2h_2^3} \qquad (1)$$

where Figure 3 illustrates the pertinent dimensions. The parameter (n) is the ratio of the Young's moduli of the substrate to that of the coating. The value of n used in this study was 14×10^6 psi/65×10^6 psi = 0.215. Equation 1 is derived from simple beam theory and is appropriate for calculating the outer surface tensile stresses in a flexure specimen providing that both the substrate and the coating are homogeneous and isotropic. This is not correct for the SiC coated C/C system investigated herein, but in lieu of a more sophisticated analysis,

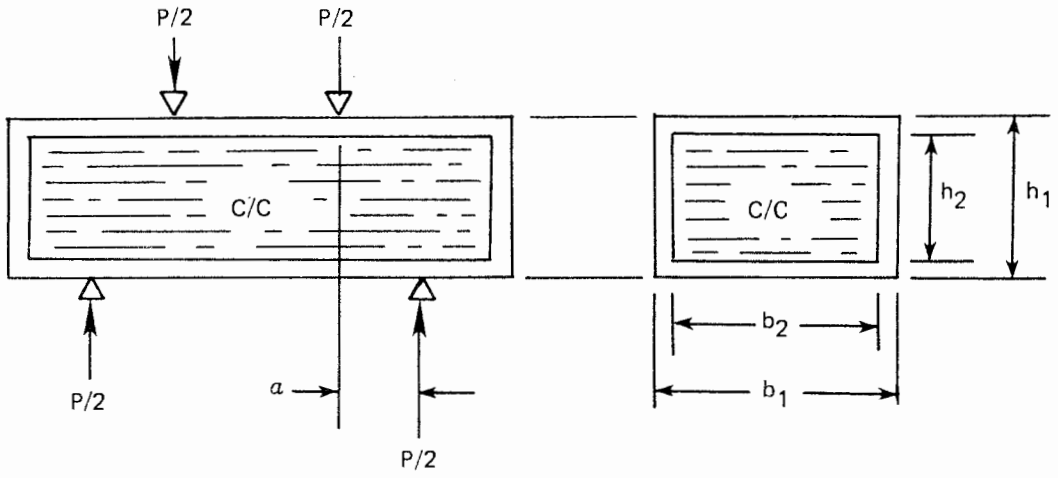


Figure 3. General schematic of the four point flexure test with applied load perpendicular to C/C plies.

5. QUINN, G. *Characterization of Turbine Ceramics After Long-Term Environmental Exposure.* U.S. Army Materials Technology Laboratory, AMMRC TR 80-15, AD A117463, April 1980.
6. QUINN, G., and KATZ, R. N. *Stepped Temperature Stress-Rupture Testing of Silicon-Based Ceramics.* Am. Cer. Soc. Bull., v. 57, no. 11, November 1978, p. 1057-1058.
7. QUINN, G., KATZ, R. N., and LENOE, E. *Thermal Cycling Effects, Stress Rupture and Tensile Creep in Hot-Pressed Si_3N_4.* Proceedings of the DARPA/NAVSEA Ceramic Gas Turbine Program Review, MCIC 78-36, August 1977, p. 715-737.
8. QUINN, G. *Guide to the Construction of a Simple 1500°C Test Furnace.* U.S. Army Materials Technology Laboratory, AMMRC TN 77-4, August 1977. Updated/revised as TR 83-1, January 1983.

Equation 1 is simply used for estimation purposes. It was further assumed for simplicity that the elastic moduli do not change appreciably with temperature. The C/C substrate maintained a significant portion of the load-carrying capacity of the composite beam. The coating and substrate thickness were measured for each bar near the point of breakage. In the limit, if the coating and the substrate had identical moduli, then n=1 and Equation 1 simplifies to:

$$\sigma = \frac{3Pa}{bh^2} \qquad (2)$$

which is the familiar formula for a homogeneous and isotropic material. Reference 4 utilizes this latter equation, an over-simplification.

Most of the room temperature flexural testing was performed with a silicon carbide fixture having 0.75" and 1.50" spans. Occasionally, a steel fixture with 0.8" and 1.6" spans was used. For consistency within this report, loads from the latter are converted to "equivalent loads" for the former when comparisons of load are made, both fixtures had 1/8"-diameter roller bearings, fixed in place, for load application.* Crosshead rate on the universal testing machine† was usually 0.20 in./min, although 0.005 in./min was occasionally employed in an attempt to discern time-dependent phenomena. A complicating matter was that surface roughness of the coated specimen (due to the nonuniformity of deposition) prevented ideal line loading in the flexure apparatus. This undoubtedly caused uneven loadings which resulted in a propensity for failure to originate at an inner load bearing. Thus, the strength values in this report have to be considered approximations, suitable for comparative value.

Throughout the remainder of this report, "strength" will be used in the context of the maximum load carrying capacity of the beam in flexure. Approximate coating stresses, as computed by Equations 1 and 2, will occasionally be reported.

All high temperature mechanical testing was performed in laboratory test furnaces as shown in Figure 4 and described in detail in Reference 8. Testing was

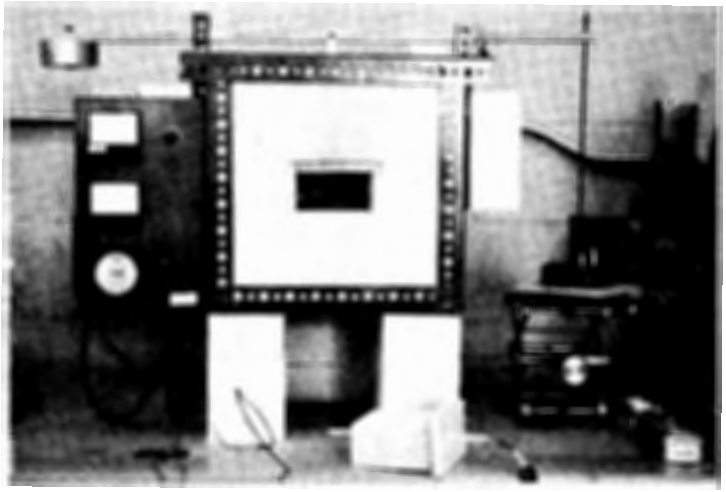


Figure 4. High temperature mechanical test furnace in the stress-rupture configuration.

*$P_{1.6} = P_{1.5}$ (0.375/0.400).
†Instron Corporation, Model TT-DL.

in an air environment at temperatures up to 2552°F (1400°C). Flexural fixtures with spans of 0.75" and 1.50" were used. A silicon carbide heating element furnace was inserted into the universal testing machine. One specimen at a time was tested in the furnace. Similar furnaces were used for stress rupture, but with deadweight lever arm-loading mechanisms as shown in Figure 4. Additional details of these experimental procedures are available in References 5 and 6.

Stepped temperature stress rupture is similar to stress rupture except that a range of temperatures are used in a stepwise sequence.[5,6] This is done to expose a stressed specimen to a wide range of temperatures to quickly assess whether there is any unusual temperature sensitivity. The sequence used in this study was to load the specimen at 1832°F (1000°C) and hold it for 24 hours, whereupon the temperature was raised 180°F (100°C) every 24 hours until the specimen failed. The final temperature was 2552°F (1400°C). Load was held constant throughout the experiment. These temperatures were chosen for consistency with earlier studies on monolithic ceramics.[5,6]

The resistance of the coated composite to thermal shock was examined two ways. First, a thermal fatigue machine was used to expose standard flexure bars on a rotating platform to an oxy-acetylene torch heating station and then to an air jet quench station. The apparatus as shown in Figure 5 is described in detail in References 5 and 7. The middle third of the bar (approximately) is heated to a temperature of 2192°F (1200°C) for two minutes. On being rotated into the air jet (which takes less than 1 sec), the specimen cooled to a black heat in 1 to 2 seconds, and room temperature in about 5 to 10 seconds. It then was incrementally rotated all the way around the carousel (taking 10 min to do so before encountering the torch station again). The carousel has six stations, so that six specimens were exposed to the thermal cycling just described. The specimens were given 100 exposures to the torch-air jet cycle and then broken in four-point bending at room temperature as described below.

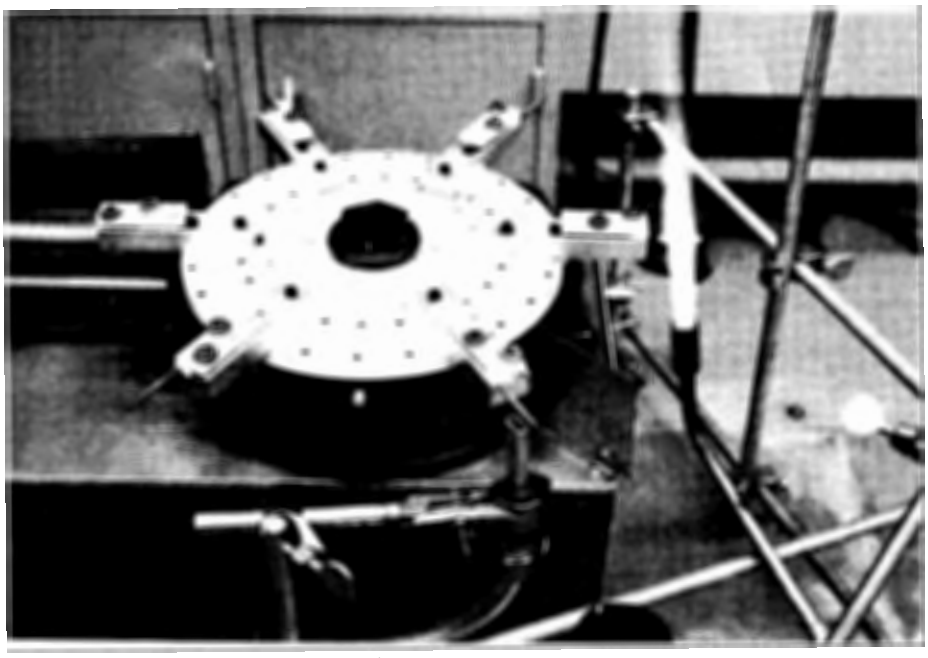


Figure 5. Thermal fatigue machine.

The second approach to discerning thermal shock damage was to heat specimens to 1868°F (1020°C) in a tube furnace and quench them once into a beaker of water at 70°F (20°C). This creates a much more severe thermal shock than the torch-air jet procedure. If the specimens survived intact, their retained flexural strength was determined.

RESULTS

Figure 6 illustrates a typical load-deflection record obtained during a fast fracture test, either at room or elevated temperature. It is not clear what the drop in load signifies during the increasing load portion of the test. It was usually accompanied by a "ping" from the specimen. A plausible explanation was that a coating crack occurred. If such a crack were large enough, it might not be "healable" and thus would represent the practical upper load bearing capacity of the coating. However, such a conclusion is premature, due to the extremely complex nature of the fracture of these specimens. None of the specimens tested with plies perpendicular to the applied load, failed in a conventional flexural mode. They all failed by crushing and spalling of the coating near one or both of the upper (inner span) load pins and deformation of the C/C composite near the load pins. In many instances, the side wall coating spalled off suggesting a buckling mode of failure. In every instance, after maximum load was achieved, the bar did not rupture, but sustained a significant load. This sequence corresponds to a fracture of the exterior coating structure, whereupon the C/C matrix is still intact and able to sustain appreciable loads.

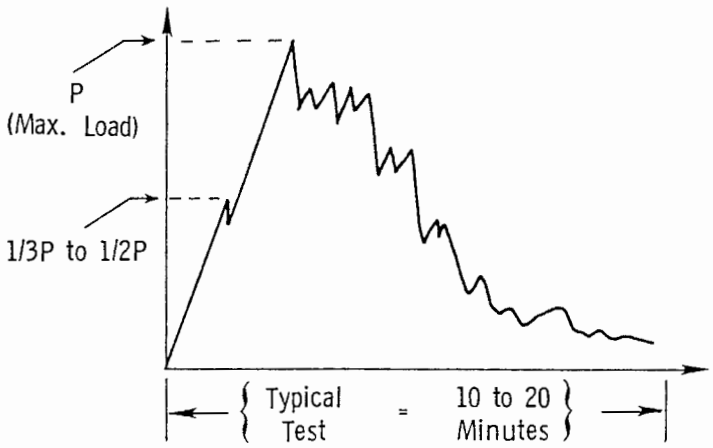


Figure 6. Typical load - deflection record during a fast fracture experiment.

Figure 7 and Table 1 give the flexure results for both coated and uncoated bars. Not surprisingly, the coated bars were stronger than the uncoated bars. The average room temperature strength of the coated specimens (39.8 lb) shall be used as a reference strength for this report. As temperature was increased, a significant strengthening occurred although the scatter also went up. Much of the original scatter in maximum load is due to coating thickness variability. Converting to maximum stress in the coating, by Equation 1 and measurements of coating dimensions, in principal this should eliminate such scatter, but Table 1 shows it does not. Caution must be exercised in the interpretation of the results in terms of stress, due to the numerous coarse assumptions implicit in Equation 1.

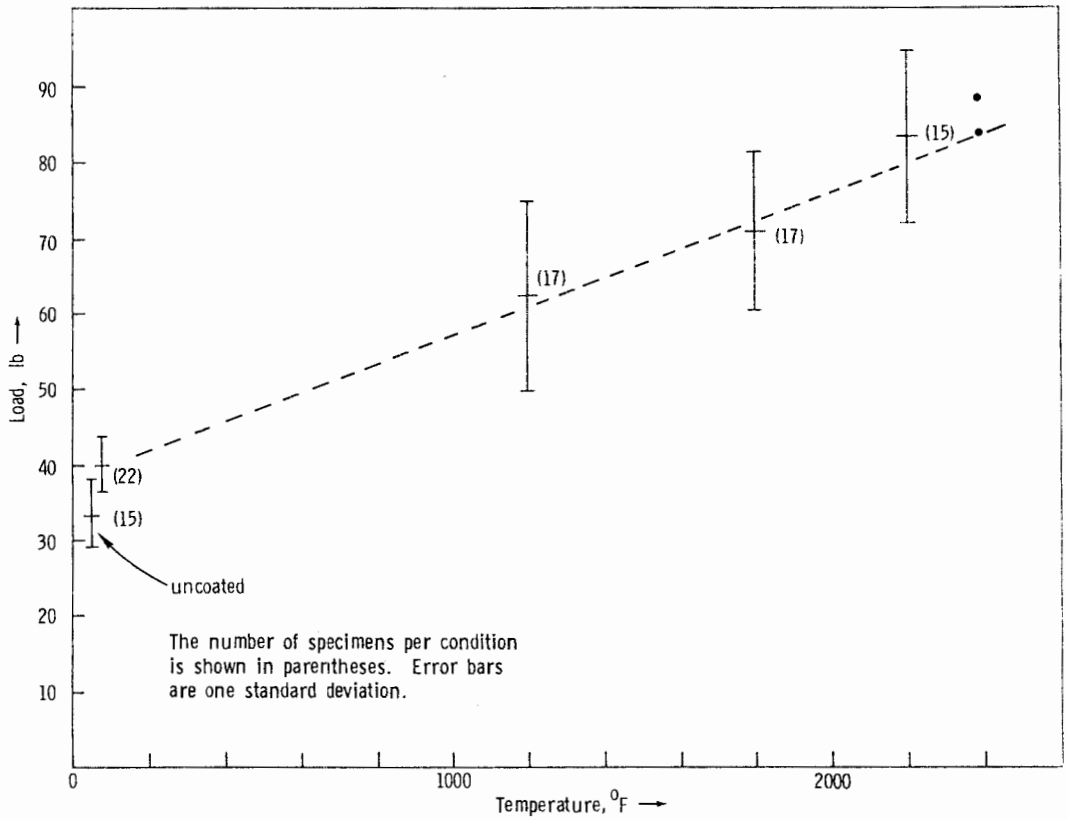


Figure 7. Flexure strength (load) versus temperature for uncoated and coated bars tested in air. The number of specimens per condition is shown in parenthesis. Error bars are one standard deviation.

Table 1. FAST FRACTURE STRENGTH OF COATED CARBON/CARBON COMPOSITE BEAMS

	Number of Specimens	Average Maximum Load* (lb)	Coating Flexure Strength, Eq. 1 (ksi)
Room Temperature (uncoated)	15	35.7 (5.0)	24.6 (3.7)
Room Temperature	22	39.8 (3.5)	36.3 (7.3)
1200°F	17	62.4 (12.5)	51.9 (14.4)
1800°F	17	70.7 (10.4)	63.5 (12.1)
2200°F	15	83.2 (11.2)	59.6 (9.2)
2400°F	2	87.8, 84.3	73.5, 76.8

*Standard deviations are in parentheses.

If for the purposes of comparison with Reference 4, the average fracture load of the reference specimens is converted by Equation 2 into an "apparent strength," the average is 23 ksi. This compares extremely well with reported results for two similar systems in Reference 4: 23 ksi for the Hitco/Ultra Carbon/SFL material and 19 ksi for the Aerojet-Bridge composite.

Figure 8 shows a typical fracture specimen, wherein the load was applied perpendicularly to the C/C plies. All of the data presented in this report are for bars tested in this fashion. Figure 9, in contrast, shows a specimen broken with the C/C plies parallel to the applied load, which resulted in a simpler fracture clearly revealing the composite beam structure.

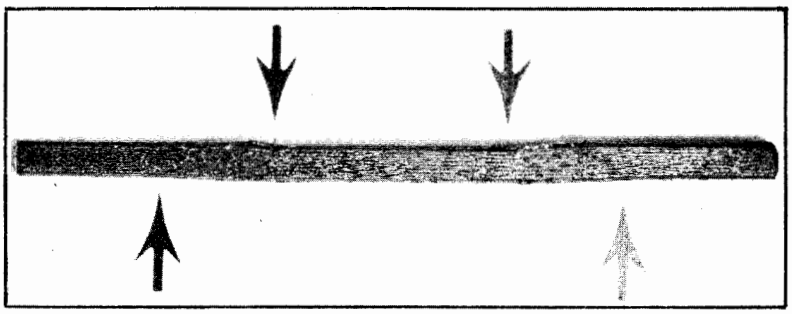


Figure 8. A coated bar after room temperature flexure testing, load applied to C/C plies. Arrows indicate load application points.

It should also be recalled that two different crosshead speeds were used: 0.02 in./min and 0.005 in./min. These crosshead speeds are not specified in the tables of data because within the range of breaking loads observed, there was no effect of crosshead speed. Most of the data (approximately 90%) was obtained using the 0.02 in./min speed. There is a final question regarding the effect of temperature on the load-bearing capabilities of uncoated C/C materials. In an inert or protected (i.e., coated) environment it might increase, however such information is not available for the substrate of interest here.

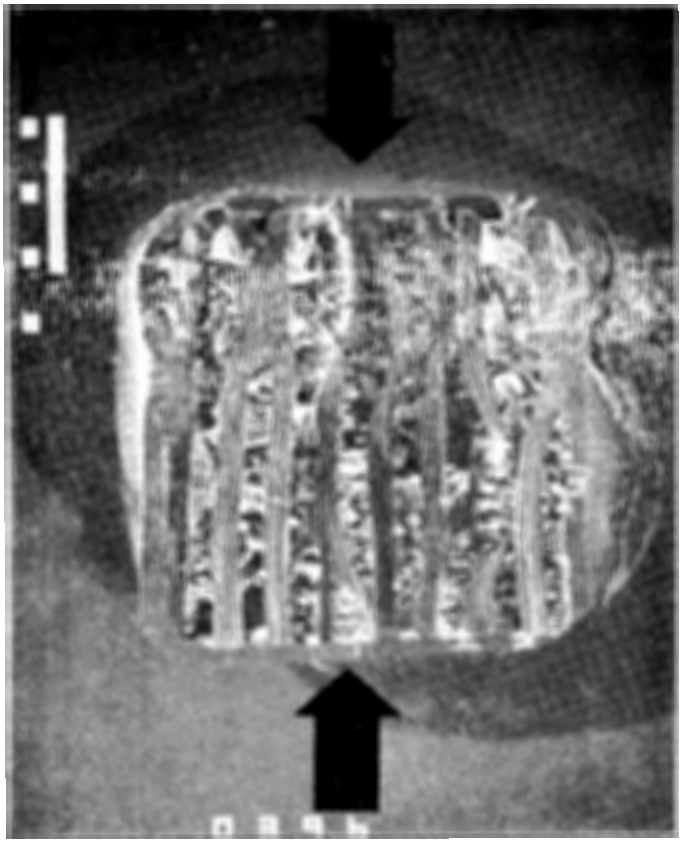
Stress-rupture results are summarized in Table 2. The five experiments at 2372°F (1300°C) all failed in a time-dependent manner, but the applied loads were equal to or higher than the average room temperature fracture load (~40 lb).

Table 2. FLEXURAL STRESS-RUPTURE RESULTS

Temperature (°C)	Flexure Load (lb)	Coating Stress	Outcome
1300	60	*	Failed < 1 hour
1300	60	*	Failed < 1 hour
1300	60	*	Failed < 1 hour
1300	60	*	Failed < 1 hour
1300	40	*	Failed at 619 hours
1200	50	+	Survived 4000 hours
1200	40	*	Failed 1.1 hours
1200	20	20 ksi	Survived 1000 hours, Retained Strength = 30 lbs
1200	20	18 ksi	Survived 1000 hours, Retained Strength = 51 lbs
1000	50	+	Survived 3000 hours
1000	50	*	Failed 2.2 hours

*Not computed because of specimen damage after fracture.

+Not broken for retained strength.

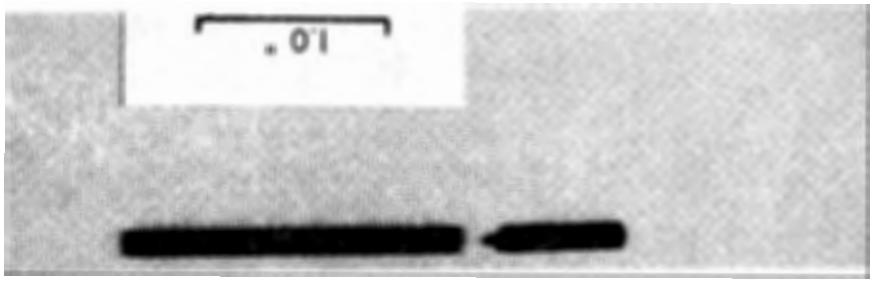


a. Fracture surface showing delaminated and pulled-out C/C plies. Direction of applied loads is indicated by arrows, bar = 1000 μm.

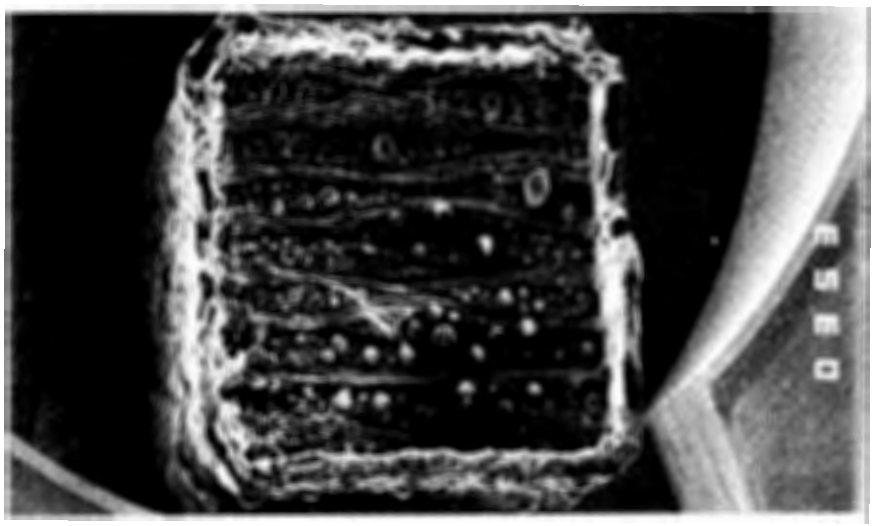
b. Corner of same specimen illustrating the duplex nature of the coating system, bar = 1000 μm.

Figure 9. A coated bar after room temperature flexure testing with load applied parallel to C/C plies resulting in a clean break through the section.

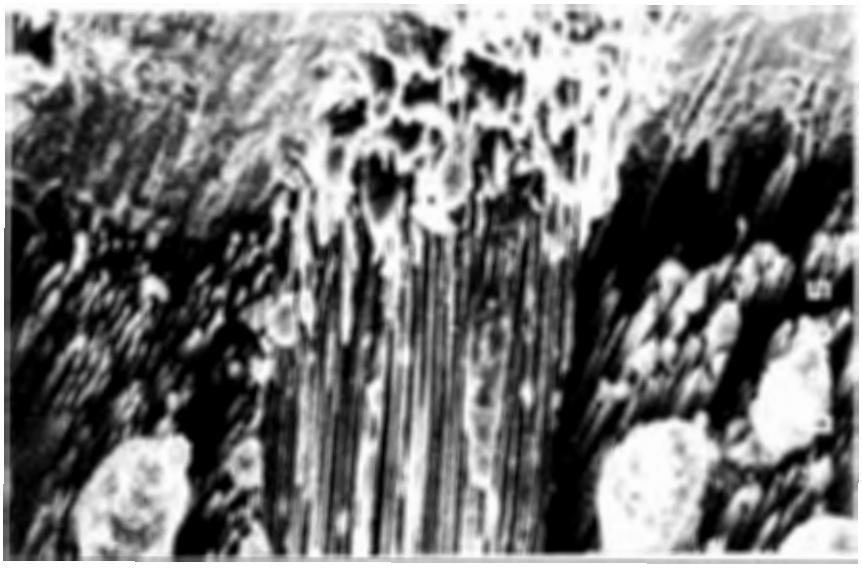
Unlike the fast fracture specimen, these did break into separate pieces.* Figure 10 shows the macro- and micro-condition of the 2372°F (1300°C) bar which failed after 618 hours. Deterioration of the C/C substrate during cooldown from the test temperature (which takes 2 to 3 hours) is evident in the two closeup views. Only two highly loaded specimens had time-dependent failure at 2200°F (1200°C) or lower. The survivors had negligible deformation (<0.1%), and had no evidence of surface degradation of any kind. The 4000-hour survivor had a weight



a. Stress-rupture specimen which failed after 618 hours at 2372°F (1300°C).



b. Cross section of the fracture surface showing deterioration of the C/C substrate, bar = 1000 μm.



c. Higher magnification photo showing the C/C ply - interlayer bonding zone, bar = 1000 μm.

Figure 10.

*This is due to the deadweight loading, unlike the conditions of a standard testing machine which applies displacement.

gain of only 0.1 percent; the 3000-hour survivor, 0.4 percent. The two 1000-hour survivors were tested at room temperature in four-point flexure with the same side and zone in tension as in stress-rupture loading. Retained strengths were not appreciably different from the reference strengths which were 31 to 45 pounds.

Three specimens were exposed to the stepped temperature stress-rupture sequence. Applied load was 40 pounds which is approximately the average reference strength. Two failed at about 1 hour at 1000°C. These had approximate (coating) stresses of 45 and 42 ksi on them at the time of failure (Equation 1). The third specimen lasted through the entire sequence, but failed while being cooled under load from 1400°C at the end of the test. It had withstood an approximate maximum (coating) stress of 37 ksi throughout the test. A slight permanent curvative was present in the specimen, the result of creep deformation. The stress rupture and stepped temperature stress-rupture experiments suggest there is no unusual temperature sensitivity in this material. The high variability in times to failure probably relates to coating thickness variability. The small permanent deformations noted in the 1200°C and 1300°C specimens are likely due to the presence of free silicon in the SiC coating. This free silicon probably accounts for the 1300°C time-dependent failures. These temperatures are near the melting point of silicon (1420°C). The influence of free silicon upon creep and crack growth in siliconized silicon carbide materials has been reported previously.[5]

The thermal shock results are summarized in Table 3. All specimens survived intact and the retained strengths are not meaningfully different from the reference strength.

Table 3.

Test	Number of Specimens	Outcome	Retained Strengths (lb)
Water Quench 1020°C to 20°C	2	Survived Intact	35.0, 42.0
Cyclic Fatigue Gas Torch - Air Blast 1200°C to 20°C	6	Survived Intact	29.0, 30.6, 32.4 34.9, 37.0, 38.8

SUMMARY AND CONCLUSION

A duplex coating system with a silicon carbide outer layer was applied by the CNTD process to flexure bars of T-300 two-dimensional weave C/C material. Earlier studies had shown that such duplex coated systems are most durable.[4] Bars were strength tested at room temperature, 1200, 1800, 2200, and 2400°F in air. Uncoated bars were also tested at room temperature. Strength measurements, either in terms of maximum load or approximate coating stress, were appreciably higher at all elevated temperatures than at room temperature. However, stress-rupture experiments showed these high strengths could not be maintained for significant times at elevated temperatures. Alternatively, several specimens with more conservative loads endured very well for thousands of hours at 1832°F (1000°C) and 2372°F (1300°C). The composite beam had favorable thermal shock resistance as demonstreated by a cyclic fatigue torch-air blast testing and also by limited water quench testing.

In general, although the coating was not uniform, was cracked, and tended to debond from the substrates, it performed its function of protecting the substrate. There was some indication that the silicate interlayer in the coating had a crack-healing capability. Further refinements to the two-layer coating process may enhance the coating/substrate bonding which may give better mechanical properties.

The laboratory testing performed in this study is of a preliminary screening nature only. No problem area was found that would rule out this composite system for advanced gas turbine engines, but much further testing is warranted. Processing work should focus upon improved coating dimensional control and uniformity. Particle impact and localized contact stress resistance should also be evaluated in order to determine if the multilayer coating will provide adequate protection from these hazards.

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