Tensile Properties of Nicalon Fiber-Reinforced Carbon Following Aerospace Turbine Engine Testing

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The durability of coated Nicalon silicon carbide fiber-reinforced carbon (SiC/C) as the flap and seal exhaust nozzle components in a military aerospace turbine engine was studied. Test specimens machined from both a flap and a seal component were tested for residual strength following extended ground engine testing on a General Electric F414 afterburning turbofan engine. Although small amounts of damage to the protective exterior coating were identified on each component following engine testing, the tensile strengths were equal to the as-fabricated tensile strength of the material. Differences in strength between the two components and variability within the data sets could be traced back to the fabrication process using witness coupon test data from the manufacturer. It was also observed that test specimens machined transversely across the flap and seal components were stronger than those machined along the length. The excellent retained strength of the coated SiC/C material after extended exposure to the severe environment in the afterburner exhaust section of an aerospace turbofan engine has resulted in this material being selected as the baseline material for the F414 exhaust nozzle system.

Keywords	ceramic matrix composites (CMCs), Nicalon silicon						
	carbide fiber (SiC/C), tensile properties, turbine en-						
	gine testing						

1. Introduction

Extensive studies are being performed by both the military and commercial engine manufacturers on advanced high temperature materials that show promise for increased engine efficiencies. One class of materials being considered for achieving more demanding thrust-to-weight goals for aerospace propulsion systems is ceramic matrix composites (CMCs). CMCs offer high specific strengths, moduli, and hightemperature capability compared with their metal counterparts. Substantial work has been performed in the area of CMCs over the last two decades, but only recently have they been demonstrated in aerospace engines.

Perhaps the most successful demonstration of CMCs in an aerospace application are variable exhaust nozzle (VEN) divergent flap and seal components on afterburning turbofan engines.^[1] The VEN is comprised of an equal number of divergent flaps and divergent seals in an axisymmetric pattern. Each seal overlaps the edges of the two adjacent flap components. A schematic representation, as given by John et al.,^[2] of the flap and seal nozzle assembly is shown in Fig. 1(a). Figure 1(b) is a photograph looking into the exhaust nozzle of one of the twin General Electric (GE) F414 turbofan engines (Cincinnati, OH) that power the Navy's F/A-18E/F Hornet fighter jet. Through relative movement of the flaps and seals, the diameter of the

gas flow path being expelled through the nozzle is adjusted. The changing diameter of the exhaust nozzle and ignition of the afterburner allows for various flight stages and maneuvers to be achieved, including supersonic flight.

To meet today's ever-increasing efficiency demands on new engines, cooling air in the exhaust nozzle is decreased and durability demands on the nozzle materials are increased. Historically, nickel-based superalloys (SAs) are used for the VEN application in military turbofan engines. However, the components made from SAs have become a source of extensive maintenance costs due to component deformation and failure under the demanding service conditions. In the exhaust nozzle, flaps and seals experience temperatures that can exceed 1000 °C,^[1] rapid heat-up and cool-down cycles, exhaust gas pressure, mechanical wear, and severe thermal gradients. The high temperatures lead to creep deformation while the thermal cycles promote cracking. An example of the damage incurred by Rene' 41 exhaust nozzle components in service is illustrated in Fig. 2. The figure shows one divergent flap surrounded by two divergent seals from a GE F110 engine that were removed following service on an F-16. The components were removed from service before reaching their design life after significant deformation and extensive cracking developed.

Early simulated mission testing on the F414 afterburner prototype showed that the metallic exhaust nozzle had inadequate life and reparability. The nozzle performance was limited by the durability of the metallic nozzle components and repairs were not cost-effective due to the single-piece component design. As an alternative to the nickel-base superalloy parts, GE and the U.S. Navy have incorporated a hightemperature CMC material into a newly modified design of the VEN on the F414 turbofan engine. The CMC is a coated Nicalon silicon carbide fiber-reinforced carbon (SiC/C). The SiC/C flaps and seals are incorporated into a removable design concept that was intended to improve the life of the components and to allow easy replacement. The flap is a flat plate that is inserted into a three-sided metal frame and backbone struc-

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Fig. 1 (a) A schematic of the VEN's axisymmetric pattern in which each seal overlaps the edges of the two adjacent $flaps^{[2]}$; (b) a view into one of the GE F414 VENs on a Navy F-18 fighter jet

ture. The backbone structures of the flaps connect to the hydraulic arms that control the size of the nozzle opening. The seal is a flat plate that has three embedded metal attachments that connect it to a backbone structure and to the flap components. An example of the components with their metal hardware is shown in Fig. 3.

Insertion of a new material and component design requires empirical verification and field testing for validation. Experiments carried out by GE Aircraft Engines on CMC hardware did show that the SiC/C material in the removable design would be a viable material for replacing the metallic nozzle hardware. They have reported that the CMC hardware has



Fig. 2 Nickel-base superalloy (Rene' 41) exhaust nozzle components removed from an F110 engine before reaching their design life due to severe deformation and cracking

shown significant benefits over the traditional metallic components. The CMCs have allowed for increased thrust through increased gas flow path temperatures and reduced cooling air requirements.^[3] The lighter SiC/C components have also been reported to provide a weight savings of about 13 pounds per engine and possibly more in total airframe weight^[4] over the original nickel-base superalloy components. However, no quantitative data on the retained strength of the SiC/C flaps and seals after service or testing on an engine are available in the literature.

It is of interest to quantitatively document the survivability or the durability of the coated SiC/C following service or testing on an F414, as the material may be susceptible to degradation if the protective coating layers are damaged. Parts handling, component installation, extreme temperatures, gas flow pressure, and the action of the nozzle widening and retracting are all potential causes of damage to the material's integrity. Therefore, this study was implemented to document the retained strength of a CMC exhaust nozzle flap and seal component after extensive time on a F414 ground-test engine.

Tension tests were performed on specimen machined from an engine tested flap and seal provided by GE Aircraft Engines (Lynn, MA). In addition, the material manufacturer provided witness coupon data from ship sets of as-fabricated flap and seal components for comparison. The durability of the engine tested SiC/C material was evaluated through a comparison of the retained tensile strengths with respect to the application requirements and to the as-fabricated material strength.

2. Material

The CMC material investigated in this study was a coated silicon carbide fiber-reinforced carbon (SiC/C) fabricated by HITCO Technologies, Inc. (now HITCO Carbon Composites, Inc., Gardena, CA) under the trademark name CeracarbSC537EH. Composite reinforcement is nanocrystalline ceramic grade NICALON silicon carbide fibers woven in a balanced eight-harness satin weave (8HSW) cloth. This



Fig. 3 F414 VEN flap (a) and seal (b) with their respective metal hardware

means fibers are woven in an "over seven, under one" sequence. The woven plies are stacked in an alternating sequence such that the warp fibers (referred to as the 0° fibers) are oriented in the axial and then the transverse direction. The stacking sequence is symmetrical about the composite centerline. There are 8 plies in the flap and 12 plies in the seal material.

The matrix contains resin based inhibited pyrolytic carbon, chemically deposited carbon, and boron carbide (B_4C) fillers. The entire composite is coated with an exterior silicon carbide coating (Chromalloy Corporation, Orangeburg, NY, RT 42, 100-150 μ m thick) and an outer coating (HITCO Technologies M185A glaze coating, 75-150 μ m thick), which provides environmental protection up to 1371 °C (2500 °F).^[5] The composite thickness is related to the number of woven plies. The flap and seal are 3.0 mm and 4.5 mm thick, respectively.

The general microstructure of the SiC/C material is illustrated in the optical micrographs of Fig. 4(a) and (b). Figure 4(a) is a low magnification representation of the transverse material cross section that shows the 0°-90° fibers, the matrix, the dual exterior coating systems, and matrix cracks that develop during processing. Figure 4(b) shows the material cross-section at higher magnification in which the B₄C filler particles, pyrolyzed carbon, and the chemically deposited carbon layers that make-up the matrix can be seen.



Fig. 4 General microstructure of the SiC/C material as manufactured by the former HITCO Technologies, Inc.: (a) at low magnification the fibers tows, the matrix, the dual exterior coating systems, and the matrix cracks from processing can be observed; (b) at higher magnification individual fibers, B_4C filler particles, and layers of chemically deposited carbon can be depicted

At the time this material evaluation study was being performed, HITCO Technologies, Inc. manufactured the flap and seal components used on the F414. The material from which the flap inserts were manufactured had eight composite plies and the seal component material, with the metal backbone structure, had 12 composite plies. The components were manufactured in multiple production runs and grouped by "ship sets." Spare components in each ship set served as "witness plates" for the manufacturing process. Three specimens machined from each witness plate were tensile tested by HITCO

Table 1Properties as Measured by HITCOTechnologies, Inc. for the 8-Ply Flap Material WitnessCoupons^[8]—the Flap Component Tested Came FromShip Set 93-4

	8-Ply Flap Material						
Ship Set No.	Avg Strength, MPa	Std Dev, MPa	Avg Modulus, GPa	Std Dev, GPa			
93-1	195.4	9.31	66.9	1.19			
93-2	200.2	13.69	72.9	2.79			
93-3	215.1	3.16	64.1	0.00			
93-4	249.4	12.81	65.3	1.05			
93-5	220.9	10.05	59.1	0.80			
93-6	190.8	8.76	62.7	3.45			
93-7	194.9	8.62	65.5	1.82			
93-8	183.9	4.89	57.2	2.07			
93-9	182.5	5.78	63.9	3.11			
93-10	209.4	4.21	67.3	1.99			
93-11	219.5	11.34	66.2	5.47			
93-12	221.3	13.10	67.6	1.82			
93-13	214.4	4.83	65.0	2.11			
	207.5	18.71	64.9	3.89			

Table 2Properties as Measured by HITCOTechnologies, Inc. for the 12-Ply Seal Material WitnessCoupons^[8]—the Seal Component Tested Came FromShip Set 93-7

	12-Ply Seal Material						
Ship Set No.	Avg Strength, MPa	Std Dev, MPa	Avg Modulus, GPa	Std Dev, GPa			
93-1	206.6	7.24	69.0	3.16			
93-2	191.2	11.48	68.5	7.75			
93-3	219.3	20.36	64.4	4.59			
93-4	237.6	8.87	62.3	2.79			
93-5	226.8		59.8	1.44			
93-6	198.8	2.87	63.7	3.98			
93-7	182.9	9.98	65.7	3.26			
93-8	205.9	6.52	66.0	2.42			
93-9	231.2	7.37	65.3	3.80			
93-10	223.2	4.19	68.5	0.80			
93-11	222.9	10.71	65.5	2.49			
93-12	215.8	17.06	66.9	3.90			
93-13							
	213.5	16.64	65.4	2.70			

for process data. The witness coupon data from several ship sets of flap and seal components were provided by the material manufacturer and are listed in Table 1 and in Table 2, respectively.^[6] The engine tested flap component provided by GE for this study came from Ship Set 93-4 (bold type in Table 1). The engine tested seal component came from Ship Set 93-7 (bold type in Table 2).

The average strengths and standard deviations for the ship sets of the 8-ply flap material ranged from 182.5 ± 5.78 MPa to 249.4 ± 12.81 MPa. The engine tested flap component came from the ship set that had the highest average strength of all the flap material production runs and one of the higher standard deviations. The average ship set strengths for the 12-ply seal material ranged from 182.9 ± 7.59 MPa to 237.6 ± 17.24 MPa. The 12-ply seal material appears to have more variability in



Fig. 5 F414 ground-test turbofan engine during operation with the integrated SiC/C flap and seal nozzle configuration^[3]

strength within a ship set of material compared with the 8-ply flap material. The engine tested seal component came from the ship set with the lowest average strength and standard deviation.

3. Experimental Procedure

The flap and seal components studied were received from GE Aircraft Engines after ground testing on a F414 afterburning turbofan engine. Ground testing consists of accelerated mission test (AMT) cycles. AMTs allow for flight maneuvers to be simulated and accumulated in a relatively short period of time compared with a fielded engine. Figure 5 shows a F414 ground-test engine with SiC/C divergent flaps and seals under afterburning conditions.^[3]

The flap and seal components that were made available for this study were tested under different AMT profiles. The flap was tested for 287 h and underwent 662 afterburner lights. The seal underwent 416 afterburner ignitions and was removed after approximately 355 h of testing due to the failure of the surrounding metal hardware. A photograph of the flap and seal components following ground engine testing is shown in Fig. 6. The damaged area on the seal is to the left in the photograph. The flap was tested for approximately 57% of its design life while the seal was tested for approximately 71% of its design life. Refer to Table 3 for a listing of the past histories of the two exhaust nozzle components.

Each component was examined for evidence of thermal and mechanical damage. In addition to the damage from the metal hardware, the seal component had very small areas where the coating had been spalled around the attachment hardware. In the spalled region the composite surface was exposed but did not appear degraded.

The flap had two large areas of surface wear that were caused by the seal rubbing against the surface of the flap. It was apparent at low magnification that the protective exterior silicon carbide coating (Chromalloy RT42) had not been worn through, but the rubbing had caused the coating to become smooth. The damage on the flap can be seen in Fig. 6. Coating

thickness measurements verified that there was no appreciable damage to the SiC coating.

Ultrasonic C-Scans were obtained from both engine components to look for internal damage and thus to verify their integrity. The C-scans were performed using a waterimmersion, pulse-echo technique in which a single transducer was used to send an ultrasonic signal through the sample and to record the amplitude of the reflected sound wave. The C-scans of the seal and flap show no indications of internal damage such as delaminations or cracks. Small deviations in the signal attenuation came from surface anomalies that could be observed visually.

3.1 Specimen Geometry

The engine tested flap and seal components were diamond cut into 100×10 mm, straight-sided test specimens. The specimens' axial direction coincided with the components' long direction. The components were divided into four rows of test specimens, namely "A, B, C, and D." Figure 7 illustrates how the components were divided into rows for test specimen machining. The machining layout for test specimens was chosen to consider differences in temperature along the length and across the width of the components.

During VEN operation, temperature gradients are set up across the width and along the length of the both the flaps and the seals. Due to the overlapping configuration of the flaps and seals, temperature gradients of approximately 200 °C can occur across the widths of the components.^[1,2,7] In the case of the flaps, the surrounding seals shield their edges making the center sections the hottest. Conversely, the seal edges are hottest where they overlap the flaps while the seal centers stay cooler. Temperature gradients along the length of the components can be up to 500 °C with the area to the aft-end of center being the hottest.^[1,2,7]

Schematics showing the specimen layout for the flap and the seal are shown in Fig. 8(a) and (b), respectively. From both components, a few specimens were machined in the transverse direction or across the component width. In the case of the flap, the specimens depicted with the open circles on the schematic (specimens F7, F16, F34, and F42) were not



Fig. 6 The divergent flap and seal components received from GE after being tested on an F414 ground-test engine

used for tension testing. These specimens contained material with small holes drilled through the thickness for thermocouple instrumentation during engine testing.

3.2 Residual Strength Testing

Residual strength tension tests were performed using a servo-hydraulic test machine with wedge grips and serrated grip inserts. The straight-sided test specimens were tabbed in the grip area with fiberglass tabs. All tension tests were run at room temperature with a displacement rate of 0.05 mm/s. A clip-gage extensometer, with a 7 mm gage length, was used to measure strain. Testing followed the guidelines identified by the American Society for Testing and Materials (ASTM).^[8]

4. Results

4.1 Engine Tested Flap

Data generated in this study for the engine-tested flap are listed in Table 4. The engine-tested results are grouped according to the row designation and labeled "A," "B," "C," and "D," accordingly. The engine-tested specimens machined in the transverse direction across the flap are shown as data set "T."

A one-way analysis of variance (ANOVA)^[9] was used to test for a difference in average strengths of the various specimen sets at a significance level of 0.05. Included in the different specimen sets are the groups of engine tested flap specimens (A, B, C, D, T) and the as-received witness flap data for ship set 93-4 (W). The calculated ANOVA F statistic for the flap data was 1.58, which has an associated probability of 0.192. Under the null hypothesis of no differences in average strengths, the critical F statistic at the 0.05 level of significance is 2.49. Since the calculated F statistic is less than the critical F statistic (the probability of observing an F of 1.58 is greater than 0.05), the null hypothesis is not rejected and the observed differences in the means are not considered statistically significant.

The conclusion from the ANOVA analysis can be observed in the plot in Fig. 9. The plot shows the residual strengths from specimens machined from the engine tested flap component and from the manufacturer's witness coupons in ship set 93-4. Each data set mean is illustrated by a dash symbol. The scatter within each data set illustrates variation in strength from specimen to specimen. The largest amount of variability is seen for the transverse specimens. Variability within the data sets makes any variation in the means small by comparison. Thus, the plot shows that the engine-tested materials appear to be

Table 3Engine Test Histories for the Flap and SealComponents Received From GE

	Total Time, h	Time at After- burner, h	After- burner Lights	Reason for Removal
Divergent Flap	287	67	662	end of test
Divergent Seal	355	22	416	metal hardware

similar in strength to each other, and to the as-fabricated materials.

4.2 Engine Tested Seal

Data obtained from the engine tested seal material are listed in Table 5. Just as for the flap specimen data, the seal data are grouped according to the axial specimen row designation or by "T" for transverse.

A one-way ANOVA^[9] was used to test for differences in mean strength among all sets of seal component specimen data, including the witness specimen data, at a 0.05 significance level. For the measured strengths, the calculated ANOVA F statistic was 31.5 and the critical F statistic was 2.60. Under the null hypothesis of no differences in average strengths, the probability of observing an F of 31.5 is less than 0.001. Since the calculated F statistic exceeds the critical value (the probability

of observing an F of 31.5 is less than 0.05) and the null hypothesis is rejected. It is concluded that at least one of the average strengths is significantly different than the others. Tukey's pair-wise comparisons^[9] indicate that the average strength for the transverse specimens was significantly greater than for the other test conditions. The average strengths for the other conditions were not significantly different.

These conclusions are somewhat obvious from Fig. 10, which includes residual strength data from the engine-tested seal component and strengths of the manufacturer's witness coupons from ship set 93-7. As with the flap data, the engine-tested results are grouped by "A," "B," "C," "D," or "T," and the as-fabricated witness coupon tensile strengths are labeled "W." Each data set mean is illustrated by a dash symbol. The plot illustrates that there are no apparent differences in mean strengths for data sets A, B, C, or D. There seems to be less overall scatter within the data sets compared with the flap



Fig. 7 Demonstrates how the flap and seal components were divided into rows for machining length-wise test specimens



Fig. 8 (a) Specimen layout used for the engine-tested flap (specimens shown with open circles were not tension tested in this study); (b) specimen layout used for the engine-tested seal

Row ID	Specimen ID	Engine Tested 8-Ply Flap Material					
		Strength, MPa	Average, MPa	Std Dev, MPa	Modulus, GPa	Average, GPa	Std Dev GPa
A	F5	258			60		
	F12	248			55		
	F14	254			58		
	F21	249	252.8	17.04	57	57.6	1.92
	F23	264			58		
	F31	265			58		
	F32	215			55		
	F39	269			60		
в	F6	257			65		
2	F11	276			62		
	F15	264			62		
	F20	254	265.8	14 61	65	61.3	4 77
	F24	248	205.0	14.01	53	01.5	4.77
	F30	295			55		
	F33	255			55		
	F38	200			62		
C	F38 E10	200			60		
C	F10 F10	292			58		
	F19 F25	209	265 1	20.84	38 64	60.9	4.15
	F23 F29	270	203.4	20.84	04 5(00.8	4.15
	F28	251			50		
D	F3/	239			00 57		
D	Fð	204			57		
	F9	265			55		
	F1/	284	0(0.4	0.71	68	50 (1.50
	F18	270	269.4	9.71	53	58.6	4.50
	F26	260			60		
	F27	257			60		
	F35	281			57		
	F36	274			59		
Т	F1	254			73		
	F2	233			67		
	F3	322			66		
	F4	318	280.5	39.66	61	61.5	6.74
	F13	308			57		
	F22	318			54		
	F40	258			60		
	F41	233			54		
		average =	266.8		average =	60.0	
		std dev =	9.93		std dev =	1.73	

Table 4 Properties Measured for the Engine Tested 8-Ply Flap Material

component data. There is, however, a large apparent variation between the axial and the transverse mean strength. When comparing the engine-tested materials to the witness coupon data, it appears the engine-tested materials are similar to the as-fabricated material and that there is no apparent loss in strength after engine testing.

5. Discussion

The room temperature residual strengths of the ground enginetested flap and seal materials were compared with the strengths of as-fabricated witness flap and seal material as reported by the manufacturer. As-fabricated material information along with engine tested materials provided a one-to-one comparison of the material's durability under simulated service conditions.

The general stress-strain behavior of the engine-tested flap and seal material is shown in Fig. 11. For comparison, a trace of as-fabricated 12-ply seal material that came from ship set



Fig. 9 Tensile strength data generated from all test specimens machined from the engine-tested SiC/C divergent flap

93-8 is shown as well. The as-fabricated data shown was generated in an earlier study by the authors.^[10] The three stress versus strain traces are very similar, however, the engine-tested materials exhibited slightly lower modulus values. The lower modulus is most likely due to an increase in the density of matrix microcracking over that present in the as-fabricated condition. This is expected due to mechanical and thermal stresses experienced in service. Even with increased matrix cracking, there is no adverse effect on the residual strength of the material. This is evidenced by comparing the residual strength to the as-fabricated material strength.

The results show that the CMC flap and seal retain their

		Engine Tested 12-Ply Sal Materia					
Row	Specimen	Strength,	Average,	Std Dev,	Modulus,	Average,	Std Dev,
	ID	MPa	MPa	MPa	GPa	GPa	GPa
А	S4	201			59		
	S5	193			56		
	S12	190			55		
	S15	194	192.9	5.52	63	60.0	6.63
	S22	183			55		
	S23	196					
	S29	193			72		
В	S 3	205			55		
_	S6	187			58		
	S11	190			57		
	S16	194	193.9	7.15	59	59.2	3.31
	S21	185			64		
	S24	200			62		
	S28	196					
С	S2	186			59		
	S 7	189			51		
	S10	188			53		
	S17	188	190.9	4.71		55.8	3.19
	S20	190			56		
	S25	198			58		
	S27	197			58		
D	S 8	189			62		
	S 9	194	191.5	2.38	55	59.0	3.61
	S18	190			60		
	S19	193					
Т	\$32	228			61		
	\$33	262	252.0	20.88	59	60.0	1.00
	S34	266			60		
		average =	204.2		average =	58.8	
		std dev =	26.74		std dev =	1.72	

Table 5 Properties Measured for the Engine Tested 12-Ply Seal Material



Fig. 10 Tensile strength data generated from all test specimens machined from the engine-tested SiC/C seal



Fig. 11 Stress-strain behavior of engine-tested flap and seal material as well as the behavior of as-fabricated 12-ply seal material^[10]

durability after the time spent on the ground-test engine. As such, temperature differences across the width of the component at locations A, B, C, and D had no significant effect on its durability. In the case of the flap material, differences in condition along the length of the component within rows A, B, C, and D might explain some of the variation within each data set. However, since the manufacturer's witness coupon data from ship set 93-4 for the flap component also shows a relatively large standard deviation in the strengths, it is likely that variability in the measured material properties is due to inherent variability in the material and or in the fabrication processes. Even so, there is a limited amount of data and no further explanation can be given at this time.

In the case of the seal material, there was indication that the orientation of the test specimen may affect the residual strength results. The transverse specimens from the seal component showed an increase in strength of up to 28%. Results for the flap were mixed, with some strength values equal to the axial specimens, and with others approximately 18% higher. Strength values in the axial and transverse directions should be similar, as this material is made with balanced 8HSW cloth and a stacking sequence symmetrical about the composite center. Steps were taken through image analysis that seemed to verify that the SiC/C material used in both the components was in fact a balanced eight-harness satin weave.

This relationship between strength and orientation has also been observed by Staehler et al.^[11] in a 0/90 woven CMC material. In that study, the axial and transverse retained tensile properties of CMC flap components were measured following ground testing on an F110 engine. In one of the Nicalon-based 8HSW balanced weave CMCs tested, SiC/SiNC, a significant difference was measured between the strengths of specimens cut lengthwise compared with those cut across the width of the flap. The transverse specimens showed nearly a 20% increase in strength compared with the axial specimens. The issue of strength variation with orientation warrants further study.

6. Conclusions

This investigation has demonstrated the robust nature of the coated SiC/C materials after being exposed to the severe ex-

haust nozzle environment during ground testing of the F414 afterburning turbofan engine. The coated SiC/C flap and seal materials tested in this study experienced no loss in tensile strength following ground engine testing equal to 57% and 71% of the respective component's service lives. Durability results such as reported in this study but generated by the engine manufacturer, has led to SiC/C being the material of choice for the F414 exhaust nozzle. In the time since this work was completed, the coated SiC/C components have been flight qualified and are now in production.

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