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FEASIBILITY OF SiC COMPOSITE STRUCTURES FOR 1644 K (2500° F) GAS TURBINE SEAL APPLICATIONS

FINAL REPORT

by

R. Darolia

November, 1979

GENERAL ELECTRIC COMPANY

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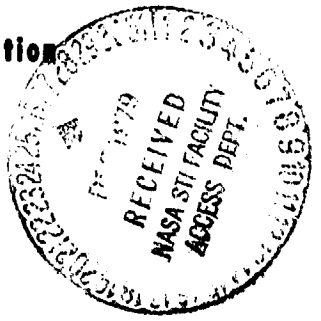

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16. Abstract

The feasibility of silicon carbide composite structures was evaluated for 1644 K (2500° F) gas turbine seal applications. The silicon carbide composites evaluated consisted of Si/SiC (Silcomp™) and sintered silicon carbide as substrates, both with attached surface layers containing BN as an additive. A total of twenty-eight candidates with variations in substrate type and density, and layer chemistry, density, microstructure, and thickness were evaluated for abradability, cold particle erosion resistance, static oxidation resistance, ballistic impact resistance, and fabricability. BN-free layers with variations in density and pore size were later added for evaluation. The most promising candidates were evaluated for Mach 1.0 gas oxidation/erosion resistance from 1477 K (2200° F) to 1644 K (2500° F). The as-fabricated rub layers did not perform satisfactorily in the gas oxidation/erosion tests. However, preoxidation was found to be beneficial in improving the hot gas erosion resistance. Overall, the laboratory and rig test evaluations have shown that material properties are suitable for 1477 K (2200° F) gas turbine seal applications. Further improvements are needed in hot gas erosion resistance and abradability to demonstrate feasibility to 1644 K (2500° F).

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FOREWORD

This Final Technical Report covers the work performed by the Aircraft Engine Group, General Electric Company, under NASA Contract NAS3-20082 and Modifications 1, 2, and 3 thereto. The NASA Project Manager was Mr. S.G. Young, Materials and Structures Division, Lewis Research Center. At General Electric, Dr. R. Darolia was the Principal Investigator and the Technical Program Manager.

The author acknowledges the efforts of the following personnel of the Materials and Process Laboratories of the Aircraft Engine Group, General Electric, who contributed to the success of the program: Mr. G.G. Wyatt and Mr. W.A. Nelson, who fabricated specimens and performed the many laboratory evaluations which were required as part of this program; Mr. R.C. Schwab and Dr. I.I. Bessen, for their valuable technical advice and counsel; Mr. J.P. Young and Mr. W.B. Foster for hot rub and high-velocity oxidation/erosion tests which played a significant role in evaluating various candidates; and finally, Dr. W.B. Hillig and Mr. C.R. Morelock of General Electric's Corporate Research and Development Center, Schenectady, who invented most of the materials evaluated in this program.

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1.0 EXECUTIVE SUMMARY

The feasibility of silicon carbide composite structures was evaluated for 1644 K (2500° F) gas turbine seal applications. The ceramic shroud materials evaluated in this program consisted of Si/SiC (Silcomp™) composite and sintered SiC substrates, both with attached surface rub layers containing BN as an additive. Later on, BN-free Si/SiC surface layers were added for evaluation. These substrates and rub layer materials were developed at General Electric's Corporate Research and Development Center. The program was accomplished in four tasks.

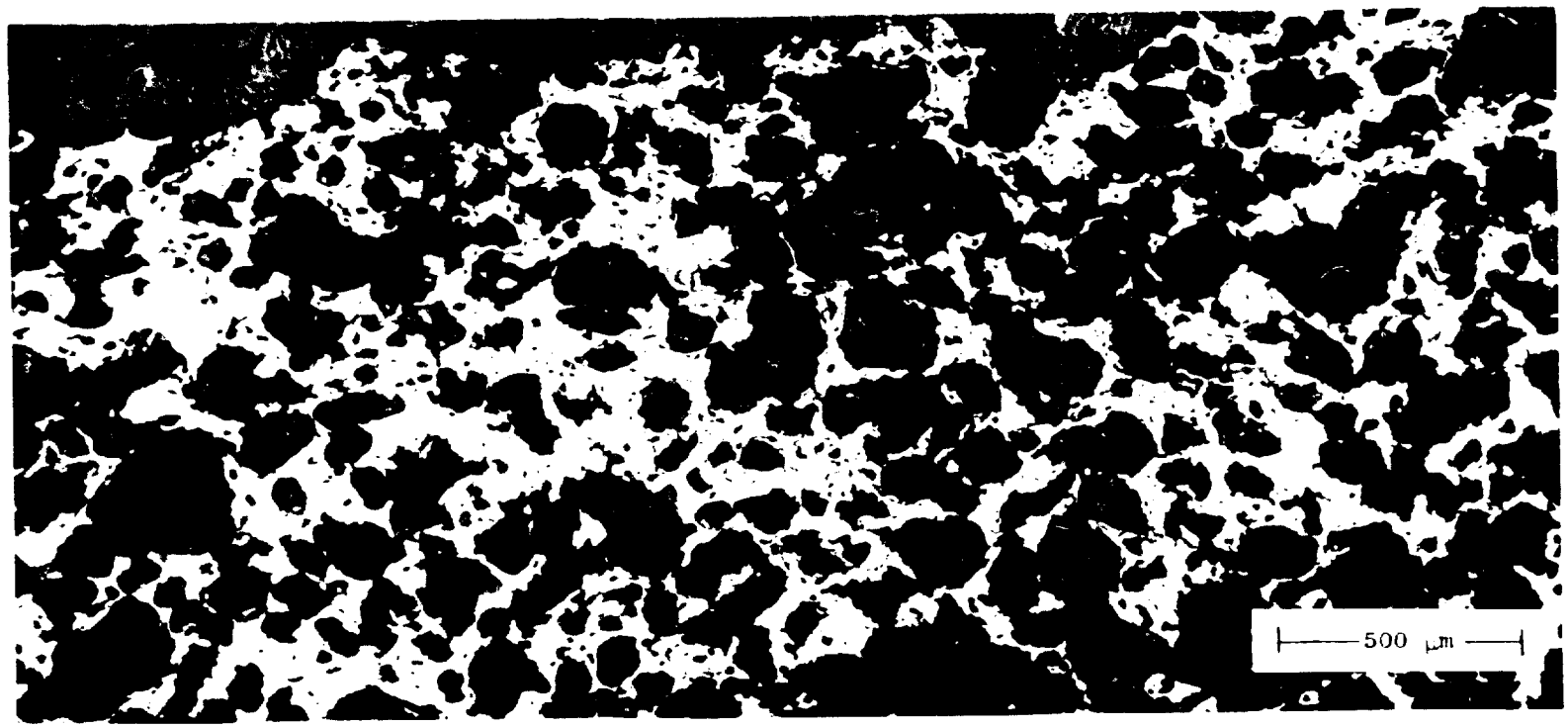
In Task I a total of 28 BN-containing rub layer candidates with variations in substrate type and density, and layer chemistry, density, microstructure and thicknesses were evaluated for abradability, cold particle erosion resistance, 1644 K (2500° F) static oxidation resistance, ballistic impact resistance, and fabricability.

In Task II nine variations of BN-free Si/SiC layer materials with variations in density and pore size were similarly evaluated. In Task III, the most promising candidates were evaluated in Mach 1.0 gas oxidation/erosion tests.

The Task I evaluation showed promise for 1644 K (2500° F) gas turbine seal application for four BN-containing ceramic shroud systems. A typical microstructure of BN-containing layers with SiC derived from graphite powder is shown in Figure 1. Figure 2 shows the microstructure of the layer attached to a Silcomp™ substrate. The BN-containing rub materials showed a marked improvement over the baseline material, Bradelloy 500, in all the laboratory evaluations. Figure 3 compares the static oxidation data of BN-containing and BN-free layer materials with Bradelloy 500.

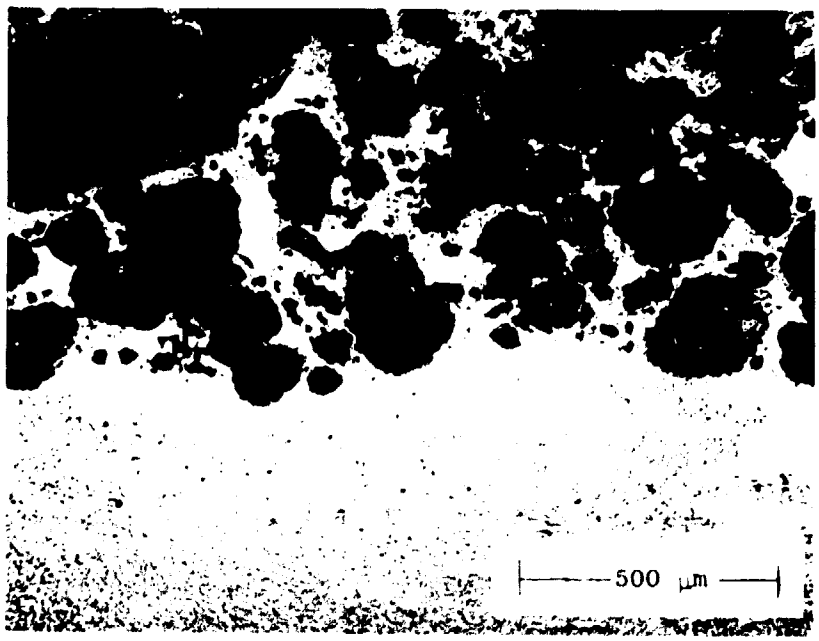
The Task II evaluations of BN-free Si/SiC rub layer materials showed that BN-free materials have better abradability, cold particle erosion resistance, 1644 K (2500° F) oxidation resistance, and ballistic impact resistance than the BN-containing Si/SiC rub layers. However, BN-free layers could not be evaluated in rig tests due to the lack of a bonding technique for attaching the layers to dense ceramic blocks. A NASA-Lewis/Solar-suggested technique which utilizes free silicon was tried and showed a good metallurgical bond in several specimens. However, the technique still needs further improvement in reproducibility and bond strength. Due to this limitation, the BN-free layers were not evaluated further.

The Mach 1.0 oxidation/erosion tests carried out in the 1477 K to 1640 K (2200° to 2500° F) range on the BN-containing systems indicated that the surface layers have adequate hot gas erosion resistance at 1477 K (2200° F). At higher temperatures, the as-fabricated layers eroded excessively. It was shown that preoxidation prior to the Mach 1.0 gas oxidation/erosion test or in the test at 1477 K (2200° F) improved the hot gas erosion resistance of the layers. The formation and retention of a silicon dioxide film around the



- Dark particles are BN grains, web network is SiC (gray), and light areas are Si.

Figure 1. Typical Microstructure of a BN-Containing Layer with SiC Derived from Graphite Powder.



- Dark particles are BN grains, grav areas are SiC, and light areas are Si (60X).

Figure 2. Typical Microstructure of Silcomp™ Substrate, Surface Layer Interfacial Area (Variation 1).

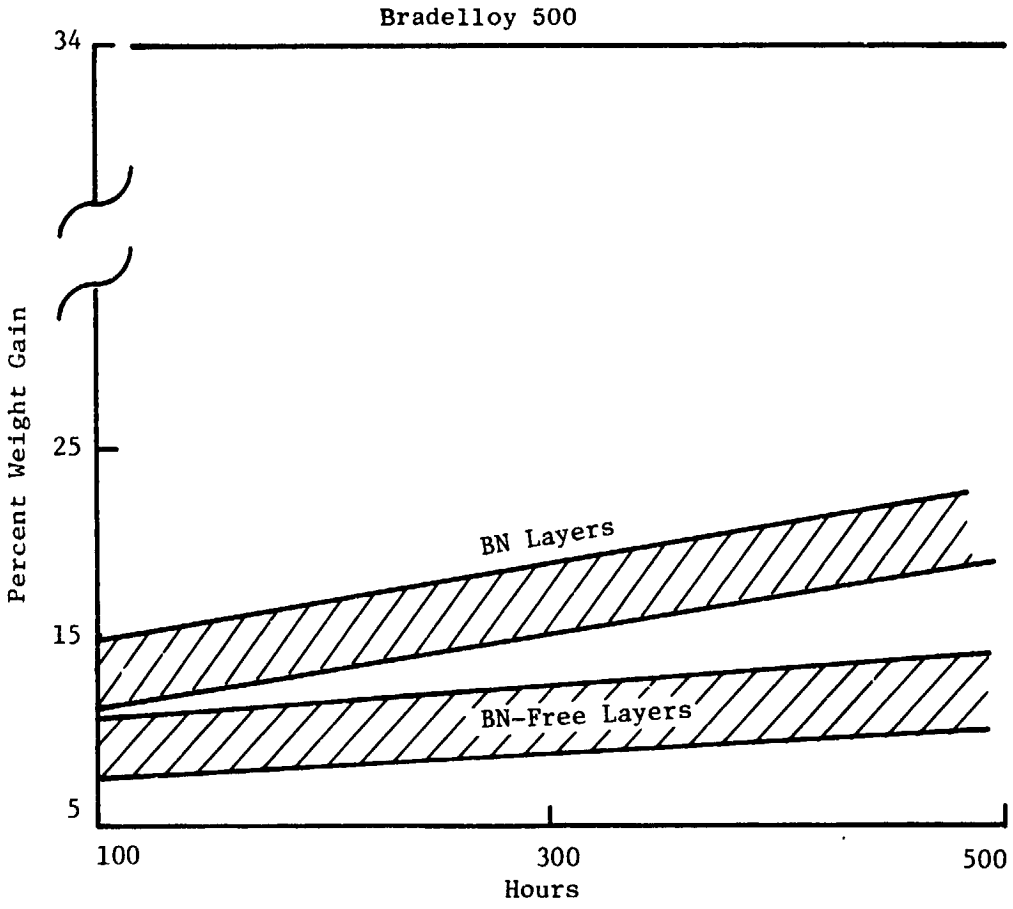


Figure 3. Typical 1644 K (2500° F) Static Oxidation Data for Bradelloy 500, BN-Containing and BN-Free Rub Layers.

Si/SiC network was found essential for good hot gas erosion resistance. Further work is needed to identify the rate and oxidation mechanism and the high-temperature retention characteristics of the silicon dioxide film. The pre-oxidation, however, reduced the abradability of the layers. Overall, a good balance between hot gas erosion resistance and abradability was not achieved.

In summary, the silicon carbide composite structures have shown good abradability and resistance to oxidation, gas erosion, thermal shock, and ballistic impact damage for up to 1477 K (2200° F) gas turbine seal applications. For 1644 K (2500° F) applications, further improvements in hot gas erosion resistance and abradability are required. In addition, the following major concerns which were not addressed in this program need to be resolved before a successful application of these ceramic materials is feasible.

1. Attachment of silicon-based ceramic components to the nickel- and cobalt-based superalloy components. The major concern is the chemical reaction between silicon of the ceramic composite and nickel and cobalt of the superalloys resulting in surface pitting on the ceramic components and the formation of low melting point silicides. In addition, better understanding of the contact stresses, point loading from machining misfit, relative motion, friction, and thermal expansion mismatch is needed.

2. Insufficient data base for reliable design. Areas to be addressed are large scatter in property data, environmental effects on the properties, and insufficient long-time property data.

Technical Plan

The technical plan for this program was divided into the following four tasks:

Task I - Material Selection and Screening

Task II - Evaluation of BN-Free Material Systems

Task III - Advanced Laboratory Evaluation

Task IV - Reporting

Task I consisted of fabrication and screening of a total of 28 variations in ceramic substrate blocks and attached ceramic surface rub layers containing BN as an additive. Two most promising variations were chosen from the initial screening for further evaluation in Task III. Task II evaluated the BN-free SiC rub layer system. The evaluation included screening nine variations to select one system. A bonding study was also undertaken to attach the BN-free rub layer to hot pressed silicon carbide and nitride. In task III the most promising systems from Task I were evaluated in Mach 1.0 gas oxidation/erosion at temperatures up to 1644 K (2500° F). A flow diagram showing the sequence of events is shown in Figure 4.

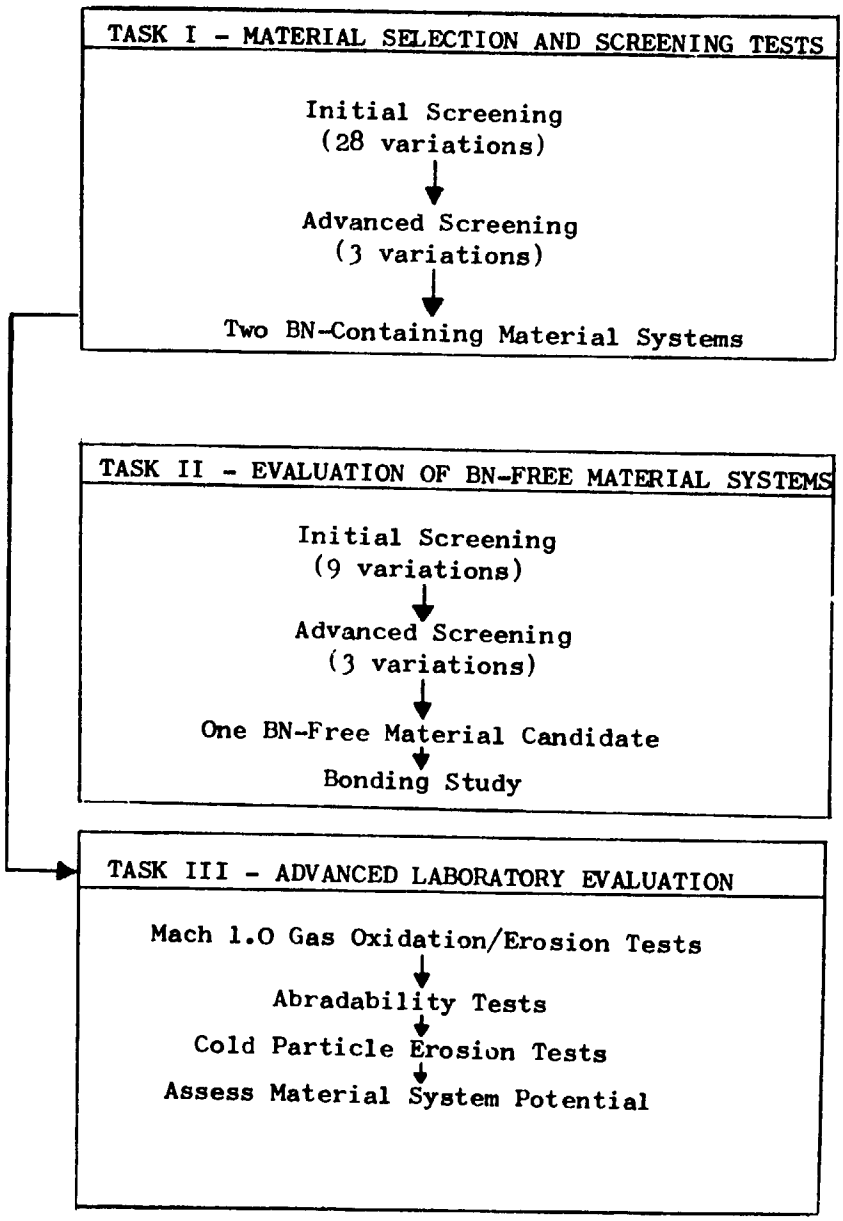


Figure 4. Flow Diagram Representing the Sequence of Events in Tasks I, II, and III.

2.0 INTRODUCTION

The demands for higher engine thrust and power output have placed new requirements on shroud materials. These requirements include: (1) shroud material temperatures of 1644 K (2500° F) and higher, (2) longer life with sustained and close clearances, (3) thermal shielding of support structures, and (4) reduction in cooling air for the shroud.

The current shroud material in General Electric commercial transport engines is Bradelloy, a low-density sintered material. The upper temperature limit for Bradelloy filler shrouds has been reached in current engines, and more oxidation resistant materials are essential. A better metallic shroud based on a NiCrAlY filler (Genaseal) was developed with support from NASA (NAS3-18905, Reference 1), and an Air Force Contract (F33615-77C-5071) is aimed at developing a lower-cost method for Genaseal. It is expected that Genaseal introduction into service within the next one to two years will accommodate longer-life needs with some improvement in temperature capability. In the 1980's even higher temperatures will be required based on the trend in advanced high-performance engine designs, and these applications will demand the use of ceramics. Ceramics with attached rub layers offer excellent oxidation resistance, the primary factor which limits metallic shrouds. A unique advantage of the ceramic surface layer is its capability to protect the substrate block from potential brittle fracture due to rotor rub or from foreign object impact.

The major objective of this contract was to determine the feasibility of silicon carbide composite structures for potential use as tip seal systems in advanced aircraft gas turbines. The expected operating seal surface temperature is 1644 K (2500° F) and above, with desired seal life in the range of 3000-4000 hours.

The ceramic shroud materials evaluated in this program included Si/SiC (Silcomp^{TM}) composite, and sintered SiC substrates, both with attached surface layers containing BN as an additive. This program was revised to include the evaluation of BN-free SiC rub layer materials and a small effort on identifying a brazing technique for attaching the BN-free SiC rub layer to a backing of hot pressed silicon carbide. These substrate and rub layer materials were developed at General Electric's Corporate Research and Development Center.

The main criteria for selection of candidate materials for the ceramic turbine shroud are high performance in the 1600 K to 1800 K (2400° to 2800° F) material temperature regime, low design risk, and low projected cost. The composite surface structure offers the best opportunity to reduce the design risk. Without the surface rub layer, metal scabs form on the plain ceramic which can act to concentrate stress and lead to fracture. The surface rub layers are expected to provide several important functions: (a) they inhibit the pickup of metal transferred from blade tips, thus avoiding the formation of scabs; (b) they have higher compliance than plain ceramics, which reduces stress concentrations both at the blade tips and the shroud surface; (c) they

increase the ballistic impact capability by an order of magnitude over plain ceramics, and (d) they provide a thermal barrier and a temperature drop between the gas path and the ceramic substrate block.

3.0 LABORATORY SCREENING PROCEDURES

The initial task (Task I Material Selection and Screening Tests) involved selection, fabrication, and screening of a total of 28 variations in ceramic substrate blocks and attached ceramic surface rub layers (Table 1). The variations included the layer/substrate block thickness ratio, type, and amount of additive in the surface rub layer, the material used for the substrate block, and the type of material used to form SiC in the surface rub layer. These 28 variations were evaluated by metallography, surface hardness measurements, cold particle erosion tests, room temperature rub tests, 1644 K (2500° F) static oxidation tests, and ballistic impact tests. These test data were compared with similar data on a baseline material, Bradelloy 500. Based on the results of the initial screening, four promising variations were further evaluated in Mach 1.0 gas oxidation/erosion tests at 1477 K (2200° F) and in 922 K (1200° F) abradability tests to select two variations for advanced laboratory evaluation in Task III.

Task II evaluated nine variations of BN-free SiC material, the variations being in the density and pore size of the material (Table 2). The nine variations were evaluated by metallography, surface hardness measurements, cold particle erosion tests, room temperature rub tests, 1644 K (2500° F) 500-hour static oxidation tests, and ballistic impact tests. Based on these preliminary screening tests, three promising variations were further evaluated in 922 K (1200° F) abradability tests to select one most promising variation. A bonding study was also made of the best BN-free abradable layer as determined by the above tests. This study included the use of high-purity silicon to join the BN-free abradable layer to a backing of hot pressed silicon nitride and hot pressed silicon carbide. The bond strengths were measured by thermal shock and mechanical strength tests and compared with the strength of the BN-containing layers.

The advanced laboratory evaluation of Task III included Mach 1.0 gas oxidation/erosion tests of the most promising systems from Task I at temperatures up to 1644 K (2500° F). Cold particle erosion, room temperature abradability, and layer surface hardness were measured before and after exposure to the Mach 1.0 hot gas. To successfully carry out this test, the high-velocity gas oxidation/erosion test holder was modified by incorporating ZrO_2 coated insulating metal tabs around the ceramic specimens to allow conducting tests up to 1644 K (2500° F) without holder deformation and without the ceramic specimen cracking due to the holding mechanism. The modified holder was checked out at temperatures up to 1644 K (2500° F) using hot pressed silicon carbide specimens without the surface layers. The screening and laboratory test details are described below.

Cold Particle Erosion Test

The cold particle erosion test is the standard test used at General Electric as a quality control check on all HPT shrouds. In this test, the weight

Table 1. Material System Variations In Task I.

Substrate	Surface Layer	Substrate Volume % SiC	Additive Level	SLT/ST
1. Cloth-Based Si/SiC	SOA BN Composition with SiC Derived from Graphite Powder	Low (SOA) (At Least 70%)	SOA	S
2. Cloth-Based Si/SiC	↓	↓	SOA	S + 0.17
3. Cloth-Based Si/SiC			SOA	S + 0.42
4. Cloth-Based Si/SiC			SOA + 5 g BN	S
5. Cloth-Based Si/SiC		↓	SOA + 10 g BN	S
6. Cloth-Based Si/SiC	↓	High	SOA	S
7. Cloth-Based Si/SiC	SOA BN Composition with SiC derived from Graphite Fiber	Low (SOA)	SOA	S
8. Cloth-Based Si/SiC	↓	Low (SOA)	SOA + 5 g BN	S
9. Cloth-Based Si/SiC	↓	Low (SOA)	SOA + 10 g BN	S
10. Cloth-Based Si/SiC	40 Mesh Prefired Porous Al_2O_3 Additive in SOA Composition (BN) with SiC Derived from Graphite Powder	Low (SOA)	Al_2O_3 Volume % Equivalent to BN Volume % in Variation 1	S
11. Cloth-Based Si/SiC	↓	Low (SOA)	Al_2O_3 Volume % Equivalent to BN Volume % in Variation 4	S
12. Cloth-Based Si/SiC	↓	Low (SOA)	Al_2O_3 Volume % Equivalent to BN Volume % in Variation 5	S
13. Cloth-Based Si/SiC	SOA BN Composition with Fine BN Particle Addition and with SiC Derived from Graphite Powder	Low (SOA)	SOA BN with X Wt % Fine Addition	S
14. Cloth-Based Si/SIC	↓	Low (SOA)	SOA BN with 2X Wt % Fine BN Addition	S
15. Cloth-Based Si/SiC	↓	Low (SOA)	SOA BN with 3X Wt % Fine BN Addition	S

SOA = State of the Art
SLT/ST = Surface Layer Thickness/Substrate Thickness Ratio
S = Standard SLT/ST

Table 1. Material System Variations In Task I (Concluded).

Substrate	Surface Layer	Substrate Volume % SiC	Additive Level	SLT/ST
16. Tow-Based Si/SiC (45°/135° TH)	SOA BN Composition with SiC Derived from Graphite Powder	Low (SOA)	SOA	S
17. Sintered, Dense SiC		---	SOA	S + 0.17
18. Sintered, Dense SiC		---	SOA	S + 0.42
19. Tow-based Si/SiC		High	SOA	S
20. Sintered, Dense SiC		---	SOA	S
21. Sintered, Dense SiC		---	SOA + 5 g BN	S
22. Sintered, Dense SiC		---	SOA + 10 g BN	S
23. Sintered, Dense SiC	SOA BN Composition with SiC Derived from Graphite Fiber	---	SOA	S
24. Sintered, Dense SiC		---	SOA + 5 g BN	S
25. Sintered, Dense SiC		---	SOA + 10 g BN	S
26. Sintered, Dense SiC	1976 SOA BN Composition with SiC Derived from Graphite Powder	---	SOA	S
27. Sintered, Dense SiC		---	SOA + 5 g BN	S
28. Sintered, Dense SiC		---	SOA + 10 g BN	S

SOA = State of the Art
SLT/ST = Surface Layer Thickness/Substrate Thickness Ratio
S = Standard SLT/ST

Table 2. BN-Free Material Variations in Task II.

Variation No.	Density, $kg/m^3 \times 10^3$	Pore Size, μm
1	1.25 ± 0.05	125
2	1.25 ± 0.05	180
3	1.25 ± 0.05	250
4	1.35 ± 0.05	125
5	1.35 ± 0.05	180
6	1.35 ± 0.05	250
7 *	1.45 ± 0.05	125
8 *	1.45 ± 0.05	180
9 *	1.45 ± 0.05	250

* Variations selected for advanced screening tests.

loss of a specimen impinged upon by alumina particles under the following conditions is used as a relative measure of the erosion resistance of the material.

- Nozzle to specimen distance = 1.27×10^{-2} m (0.5 in.)
- Angle of particle approach = 90°
- Al_2O_3 powder particle size = 50μm
- Pressure = 2.07×10^5 Pa (30 psi)
- Duration of test = 45 sec
- Nozzle bore diameter = 4.78×10^{-3} m (0.188 in.)
- Amount of Al_2O_3 = 0.25 kg

This test was used only as an aid in ranking the materials, and the absolute loss measured was not used as an acceptance or rejection criterion.

Room Temperature Rub Test

Room temperature rub tests (Figure 5) were carried out at the following conditions:

No. of blades	15
Blade material	René 80
Blade tip speed	2.29×10^2 m/s (750 ft/sec)
Depth of incursion	3.81×10^{-4} m (0.015 in.)
Incursion rate	2.54×10^{-5} m/s (0.001 in./sec)

The amount of blade wear was determined for each test. In addition, visual examinations of the blade tip and the shroud surface layer were made to determine blade material transfer (scabbing), layer smearing, and blade distress such as bending, burring, and breaking. The purpose of the rub test was to rank the materials as to their rub behavior and to obtain an assessment of scabbing tendency and blade wear. Scabbing should be avoided because it promotes rapid, nonuniform blade wear resulting in loss of turbine clearances. In ceramic composite structures, scabbing can cause fracture of the substrate block by transmitting high rub forces to the block.

Surface Hardness Measurements

Surface hardness was found to correlate well with rub behavior in the surface rub layers. Consequently, hardness measurements were added to the evaluations. The hardness scale was selected as R_{15Y} which uses a 1.27×10^{-2} m (0.5 in.) diameter steel ball indentor and a load of 15 kg.

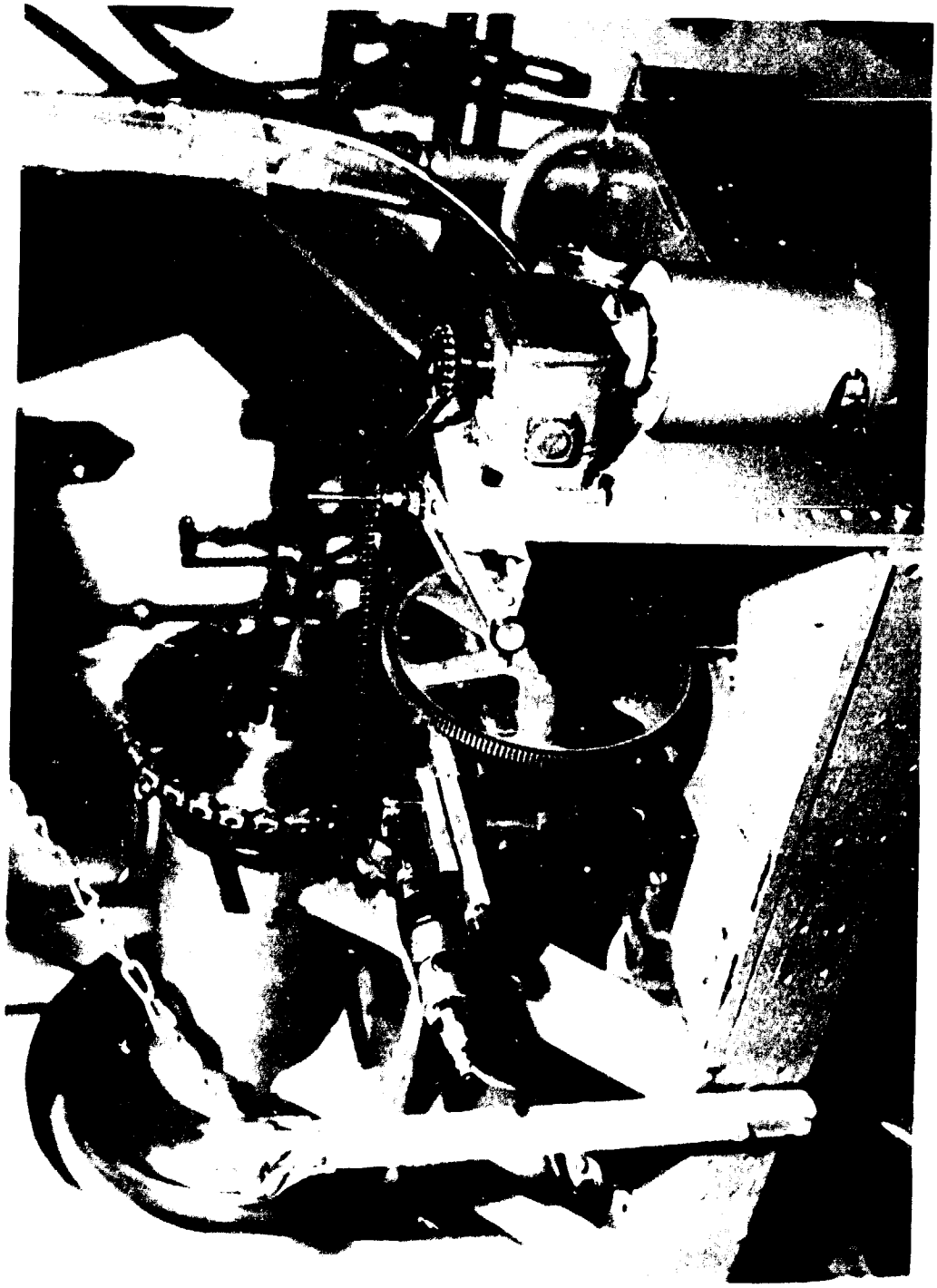


Figure 5. Abradability Tester.

Static Oxidation Test

The oxidation exposure test was performed in a resistance heated furnace. Samples were heated to 1644 K (2500° F) for up to 500 hours in a static air atmosphere.

Ballistic Impact Test

The test uses an air rifle to propel a steel pellet of 4.45×10^{-3} m (0.175 in.) diameter weighing 0.36 grams. A pellet velocity of 1.95×10^{2} m/s (640 ft/sec) was required to give an impact energy of 6.8 J (5 ft-lbf). The specimens were examined visually and by a fluorescent penetrant inspection technique.

Hot Rub Tests

Hot rub tests were conducted at the following conditions:

Temperature	877 K to 955 K (1120° to 1260° F)
No. of blades	48
Blade material	IN625
Blade thickness	6.35×10^{-4} m (0.025 in.)
Rate of incursion	2.54×10^{-5} m/s (0.001 in./sec)
Depth of incursion	6.86×10^{-4} m to 1.04×10^{-3} m (0.027 to 0.041 in.)
Blade tip speed	1.52×10^{2} m/s (500 ft/sec)

The temperature was recorded by a thermocouple placed in the surface layer. Rub forces were measured by using a load cell. At the end of each test the amount of blade wear and the depth of rub were determined. In addition, visual examinations of the blade tip and the specimen were made for evidence of blade material transfer (scabbing), layer smearing, and blade distress. R'80 is a commonly used blade material. IN625 was used instead of R'80 as the blade material in this test because the flow stress of IN625 at 923 K (1250° F) is equivalent to the flow stress of R'80 at 1311 K (1900° F).

Mach 1.0 Gas Oxidation/Erosion Test

The tests were carried out at the following conditions:

Gas Velocity	= Mach 1.0
No. of specimens per holder	= 6

Specimen holder rotation speed	= 350 to 400 rpm
Cycle	= 1/hour (55 minutes hot, 5 minutes cold)
Hot temperature	= 1477 K to 1644 K (2200° to 2500° F)
Cold temperature	= 422 K to 477 K (300° to 400° F)
Type of fuel	= JP-4

The temperature was monitored optically. Cycling was achieved by dropping the specimen holder from the up position in the hot gas stream to the down position in a blast of ambient air. The test determines oxidation, gas erosion, and thermal shock resistance. Since specimens were cycled once/hour in front of a high-velocity gas jet, a substantial thermal shock was introduced to the specimen. Figure 6 shows the assembled test holder with the test specimens.

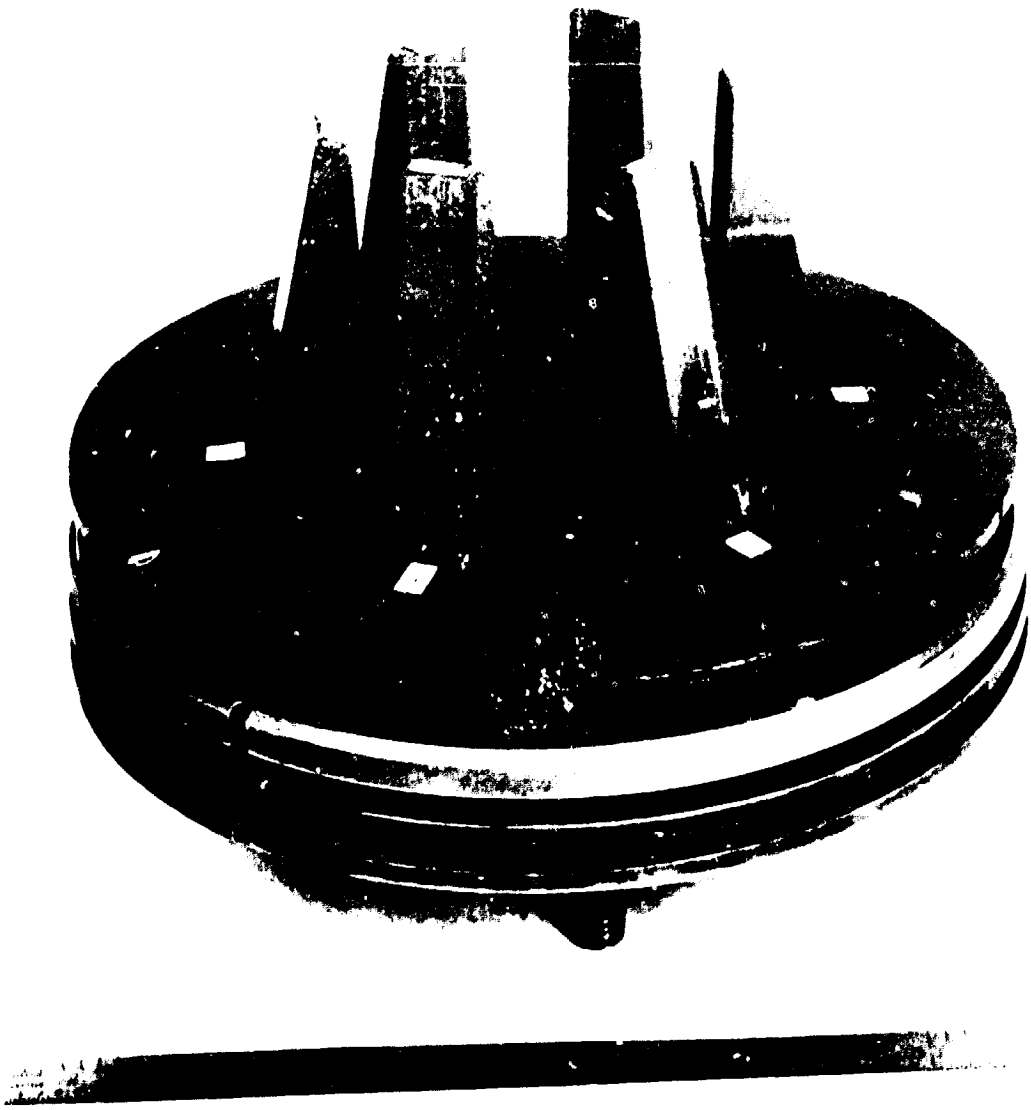


Figure 6. Mach 1.0 Oxidation Test Specimens Assembled in the Holder.

4.0 RESULTS

4.1 TASK I - MATERIAL SELECTION AND SCREENING TESTS OF BN-CONTAINING LAYER SYSTEMS

The evaluations were completed on the 28 variations in the following order:

1. Metallographic examination (Figures 1, 2, and 7 through 10)
2. Surface hardness measurement
3. Cold particle erosion test
4. Room temperature rub test
5. 1644 K (2500° F) static oxidation test
6. Ballistic impact test
7. Cold particle erosion test

In all tests, Bradelloy 500 specimens were also evaluated for comparison.

Room Temperature Rub Test

The results of the rub test are shown in Table 3. None of the variations except Variation 13 showed any blade material transfer (scabbing). In comparison, Bradelloy 500 showed scabbing and the highest blade wear/depth of incursion ratio (0.36). The blade wear/depth of incursion ratio for ceramic layers ranged from 0 to 0.29.

1644 K (2500° F) - 500-Hour Oxidation Test

The static oxidation exposure at 1644 K (2500° F) for up to 500 hours did not cause any gross melting of the substrate blocks or the layers. Also, this exposure did not cause spallation of the layers from the substrate blocks. However, some visual degradation occurred as evidenced by (1) small bubbles (0.5 mm to 2 mm diameter) coming out of the layer, (2) a glassy substance coming out of the substrate/layer interface, (3) a glassy layer on the sides of both the Silcomp™ and the sintered SiC, and (4) very small bubbles (up to 0.2 mm diameter) at the sides of the sintered SiC. X-ray diffraction analysis showed the bubbles (1 and 4) to be silicon with a small amount of the lower temperature form (tetratgonal) of cristobalite (SiO_2), and (2) and (3) to be the lower temperature form of cristobalite. The oxidation exposure also caused oxidation of the BN additive which vaporized at the test temperature, leaving holes (voids) in the layer structure (Figure 11).

- Dark particles are BN grains, gray areas are SiC, and light areas are Si (70X).

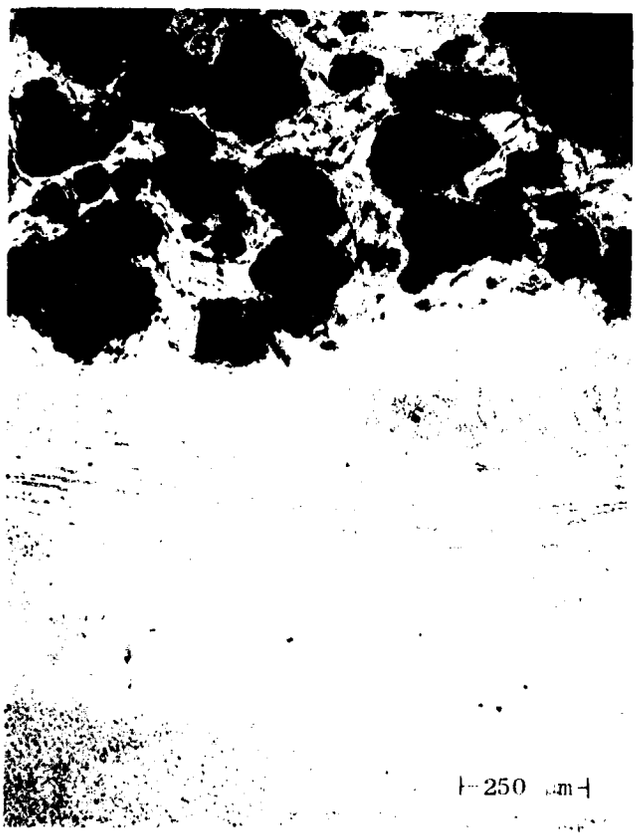


Figure 7. Typical Microstructure of Sintered SiC Substrate - Surface Layer Interface Area (Variation 20).

- Gray areas are SiC and light areas are Si (50X).

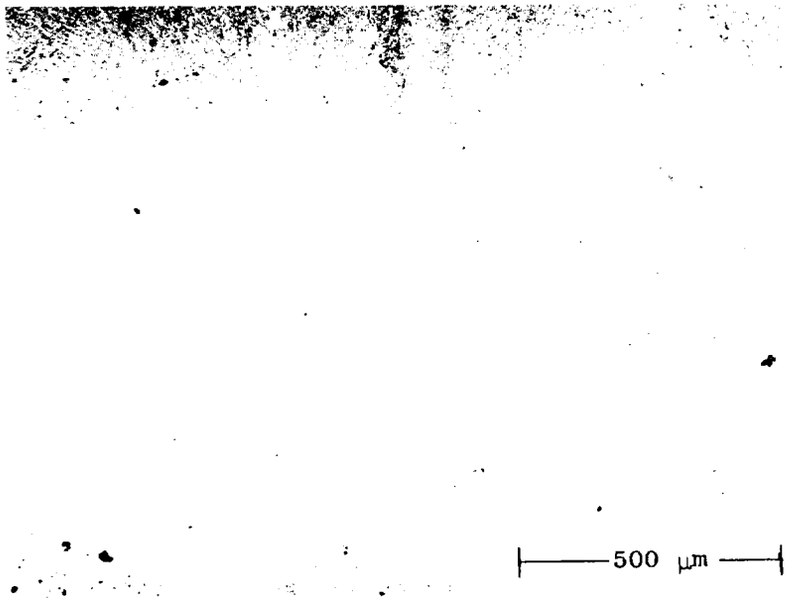
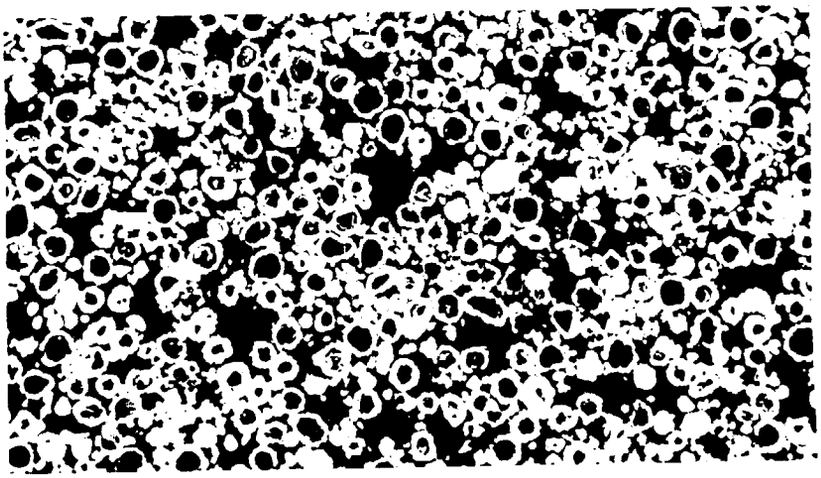


Figure 8. Typical Microstructure of Cloth-Based Silcomp™ Substrate Block with High Volume Percent SiC (Variation 6).

Figure 9. Typical Microstructure of Sintered SiC.



50X

Figure 10. Typical Microstructure of Bradelloy 500.

Table 3. Room Temperature Rub Test Data on BN-Containing Material Systems.

Specimen Variation No.	Depth of Incursion m x 10^{-4}	(mils)	Blade Wear m x 10^{-5}	(mils)	Blade Wear / Depth of Incursion	Scab
1	3.6	(14)	10	(4.0)	0.28	No
2	4.1	(16)	6.4	(2.5)	0.16	Some smear
3	4.1	(16)	8.4	(3.3)	0.21	No
4	4.1	(16)	10	(4.1)	0.26	No
5	4.1	(16)	0	0	0	No
6	4.1	(16)	1.3	(0.5)	0.03	No
7	3.8	(15)	0	0	0	No
8	3.8	(15)	2.5	(1.0)	0.07	No
9	3.8	(15)	1.3	(0.5)	0.03	No
10	3.6	(14)	0	0	0	No
11	4.1	(16)	0	0	0	No
12	3.9	(16)	0	0	0	No
13	4.3	(17)	13	(5.0)	0.29	Yes, broke two blades
14	4.1	(16)	2.5	(1.0)	0.06	No
15	4.1	(16)	7.1	(2.8)	0.18	No
16	3.8	(15)	2.5	(1.0)	0.06	No
17	4.1	(16)	6.4	(2.5)	0.16	No
18	4.1	(16)	7.6	(3.0)	0.19	No
19	4.1	(16)	3.3	(1.3)	0.08	No
20	4.2	(17)	3.3	(1.3)	0.08	No
21	4.2	(17)	2.8	(1.1)	0.07	No
22	4.1	(16)	1.9	(0.8)	0.05	No
23	4.1	(16)	2.5	(1.0)	0.06	No
24	4.1	(16)	2.8	(1.1)	0.07	No
25	3.8	(15)	0.41	(0.2)	0.01	No
26	3.9	(16)	9.7	(3.8)	0.25	No
27	4.1	(16)	2.5	(1.0)	0.06	No
28	3.8	(15)	2.5	(1.0)	0.07	No
Bradelloy						
500	4.3	(17)	15.0	(6.1)	0.36	Yes, material pull out

- The gray areas are voids where additive grains have been surrounded by an SiC + SiO_2 network.

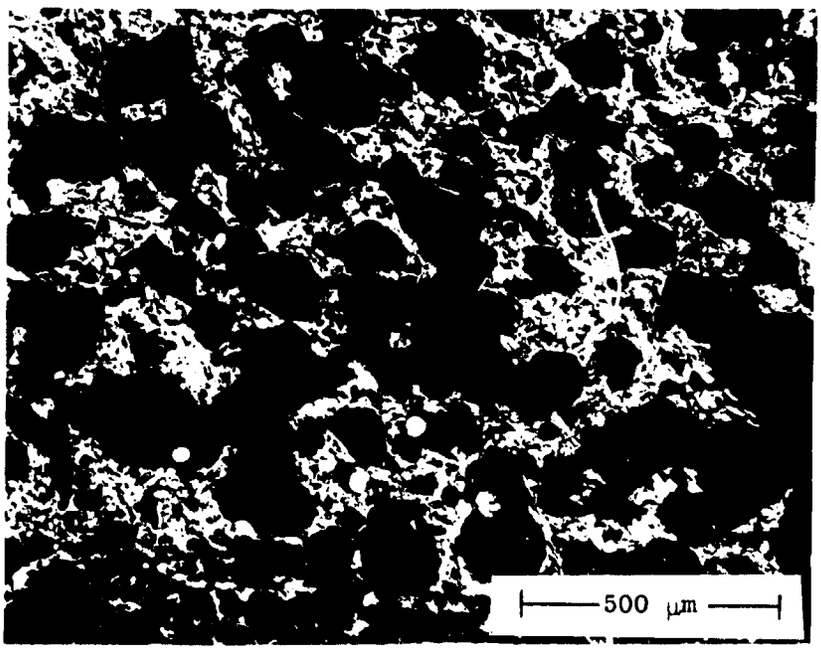


Figure 11. Typical Microstructure of a Cross Section of Rub Layer Variation 26 After Static Oxidation Exposure at 1644 K (2500° F) for 500 Hours.

The foliowing general conclusions were drawn from the oxidation data (Figures 12 through 17).

1. The oxidation exposure caused weight and volume gains in all 28 variations.

2. The variations with thicker layers (Nos. 2, 3, 17, and 18) showed larger weight gains indicating the oxidation rate of the layer is higher than that of the substrate block.

3. The variations with the sintered SiC blocks with standard SOA* layers (Nos. 20, 26) showed larger weight gains compared to cloth-based Silcomp™ blocks with similar layers (Nos. 1, 6). The initial oxidation of free silicon on the surface of Silcomp™ provides a surface sealing effect which probably slows down further oxidation. The density of the sintered SiC blocks used in this program was about 96% of theoretical, and the higher weight gains may be due to the presence of surface pores.

4. The weight gains were larger in layers containing higher BN additive levels (Nos. 4, 5, 21, 22, 27, 28).

5. Bradelloy 500 was completely oxidized when inspected after 100 hours of exposure 1644 K (2500° F). Figure 18 summarizes the oxidation data for the BN-containing layer systems and Bradelloy 500.

Surface Hardness Measurements

Surface layer hardness measurements taken on each variation in the as-fabricated condition are shown in Table 4. The correlation between surface layer hardness and rub behavior of the layers in the as-fabricated condition is shown in Figure 19. The surface hardness of a ceramic rub layer can be related to the crushing strength of the layer by the following empirical relationships:

$$\sigma = \frac{\text{LOAD}}{2\,R\,\frac{(100 - \text{Hardness})}{1000}}$$

where σ = crushing strength in kg/mm^2

R, radius of the indentor = 6.35 mm

Load = 15 kg

Hardness = R_{15Y} scale

*SOA = State of the art

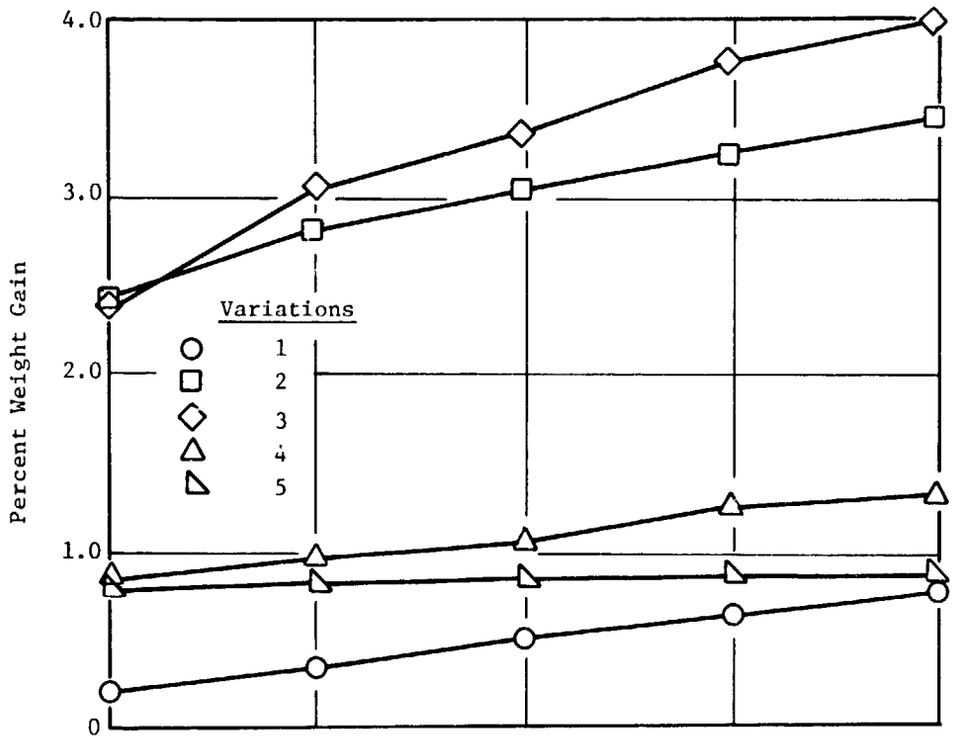


Figure 12. 1644 K (2500° F) Static Oxidation Data for BN-Containing Layer Specimens (Variations 1 Through 5).

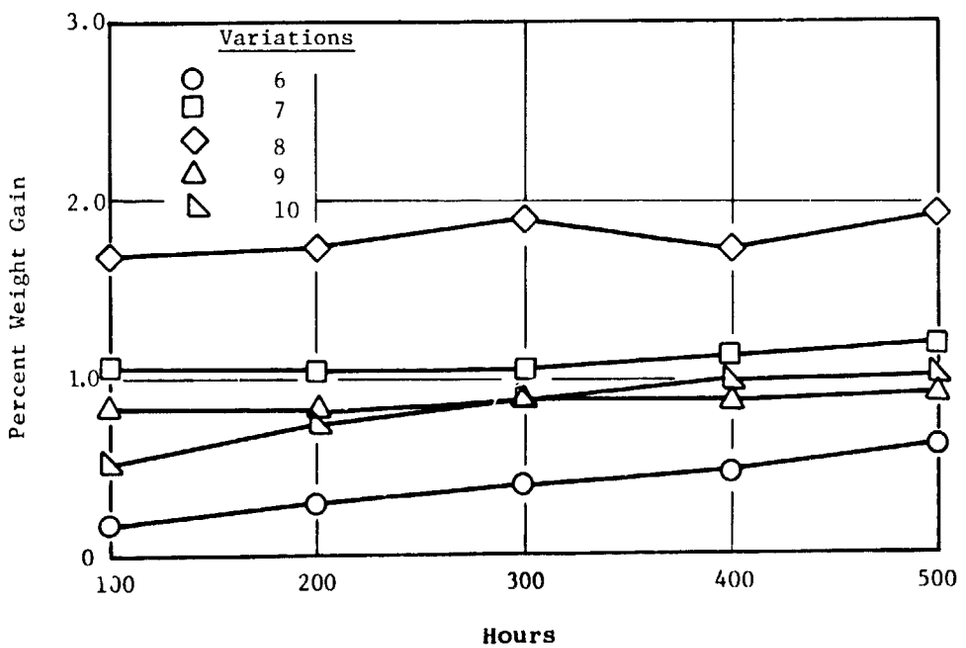


Figure 13. 1644 K (2500° F) Static Oxidation Data for BN-Containing Layer Specimens (Variations 6 Through 10).

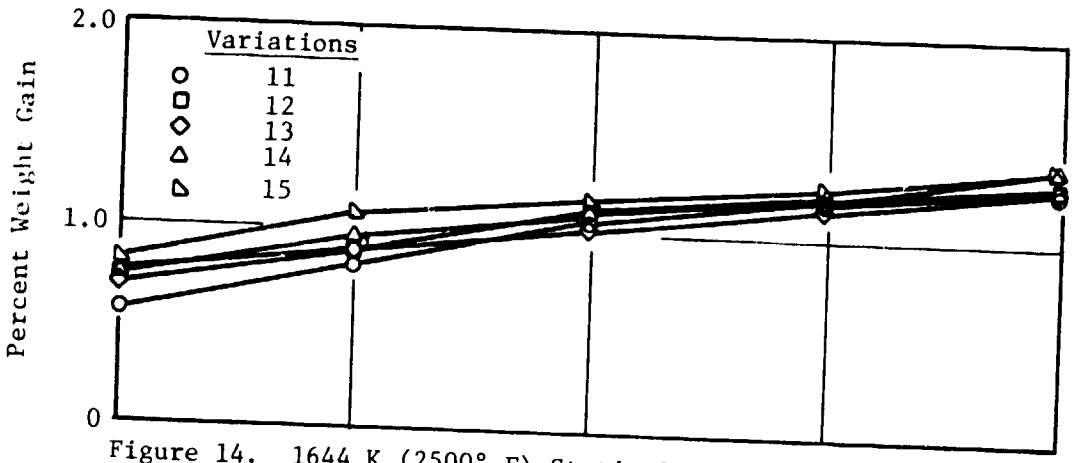


Figure 14. 1644 K (2500° F) Static Oxidation Data for BN-Containing Layer Specimens (Variations 11 Through 15).

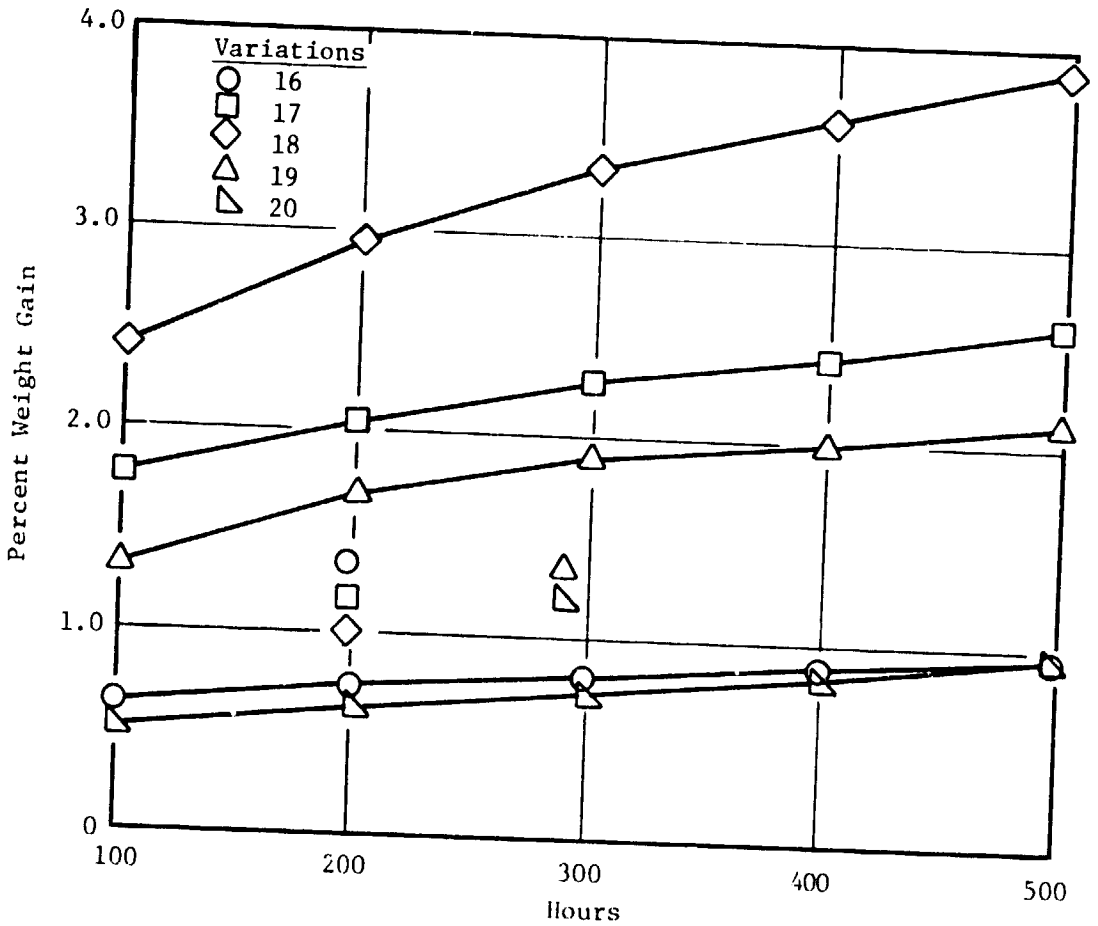


Figure 15. 1644 K (2500° F) Static Oxidation Data for BN-Containing Layer Specimens (Variations 16 Through 20).

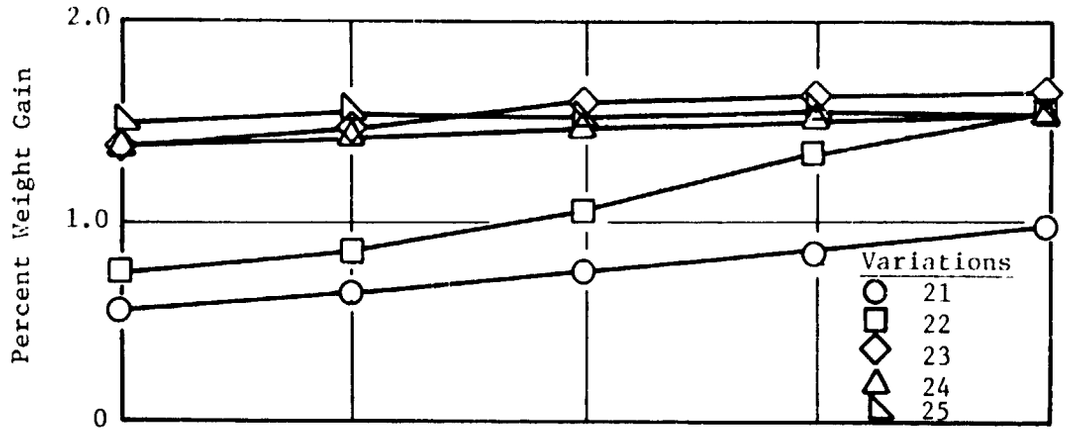


Figure 16. 1644 K (2500° F) Static Oxidation Data for BN-Containing Layer Specimens (Variations 21 Through 25).

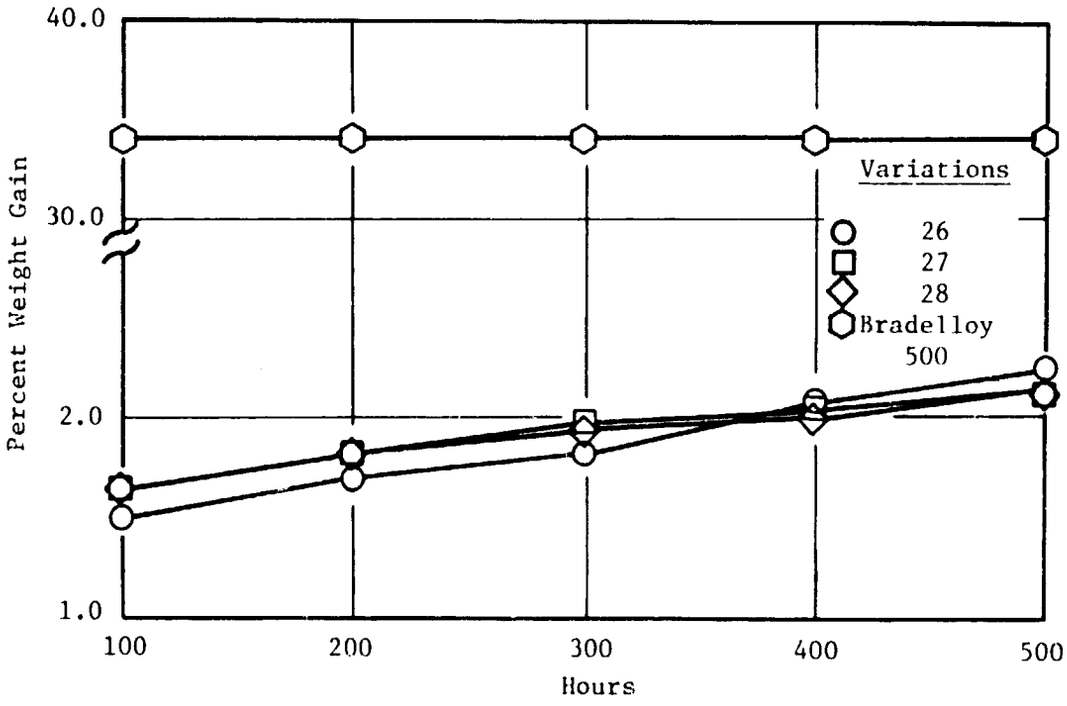


Figure 17. 1644 K (2500° F) Static Oxidation Data for BN-Containing Layer Specimens (Variations 26 Through 28) and Bradelloy 500.

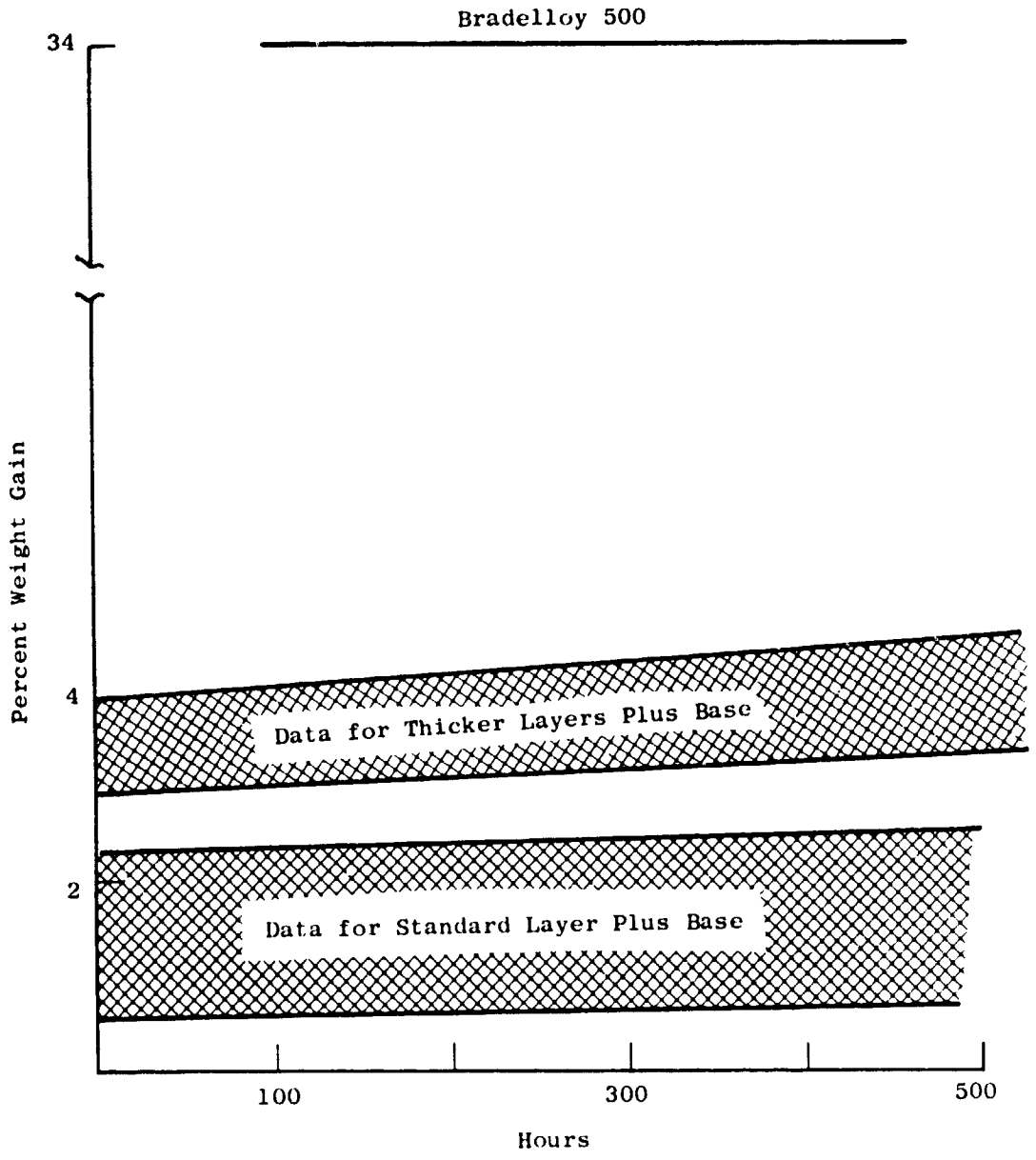


Figure 18. Summary of 1644 K (2500° F) Static Oxidation Data for BN-Containing Layer Specimens and Bradelloy 500.

Table 4. Surface Hardness Data on BN-Containing Material Systems.

(Average of Six Readings)

Specimen Variation No.	As-Processed Hardness, R_{15Y}	After 1644 K (2500° F) 500-Hr Exposure Hardness, R_{15Y}
1	73	81
2	63	77
3	57	76
4	65	85
5	3	6
6	0*	29
7	0	0
8	7	36
9	1	21
10	0	28
11	0	0
12	0	0
13	88	87
14	74	84
15	83	88
16	54	80
17	75	81
18	70	71
19	76	85
20	66	78
21	53	80
22	47	85
23	12	61
24	27	55
25	10	20
26	66	92
27	38	79
28	27	80
Bradelloy		
500	65	0**

* Very soft, goes beyond scale

**Oxidized, beyond measurement

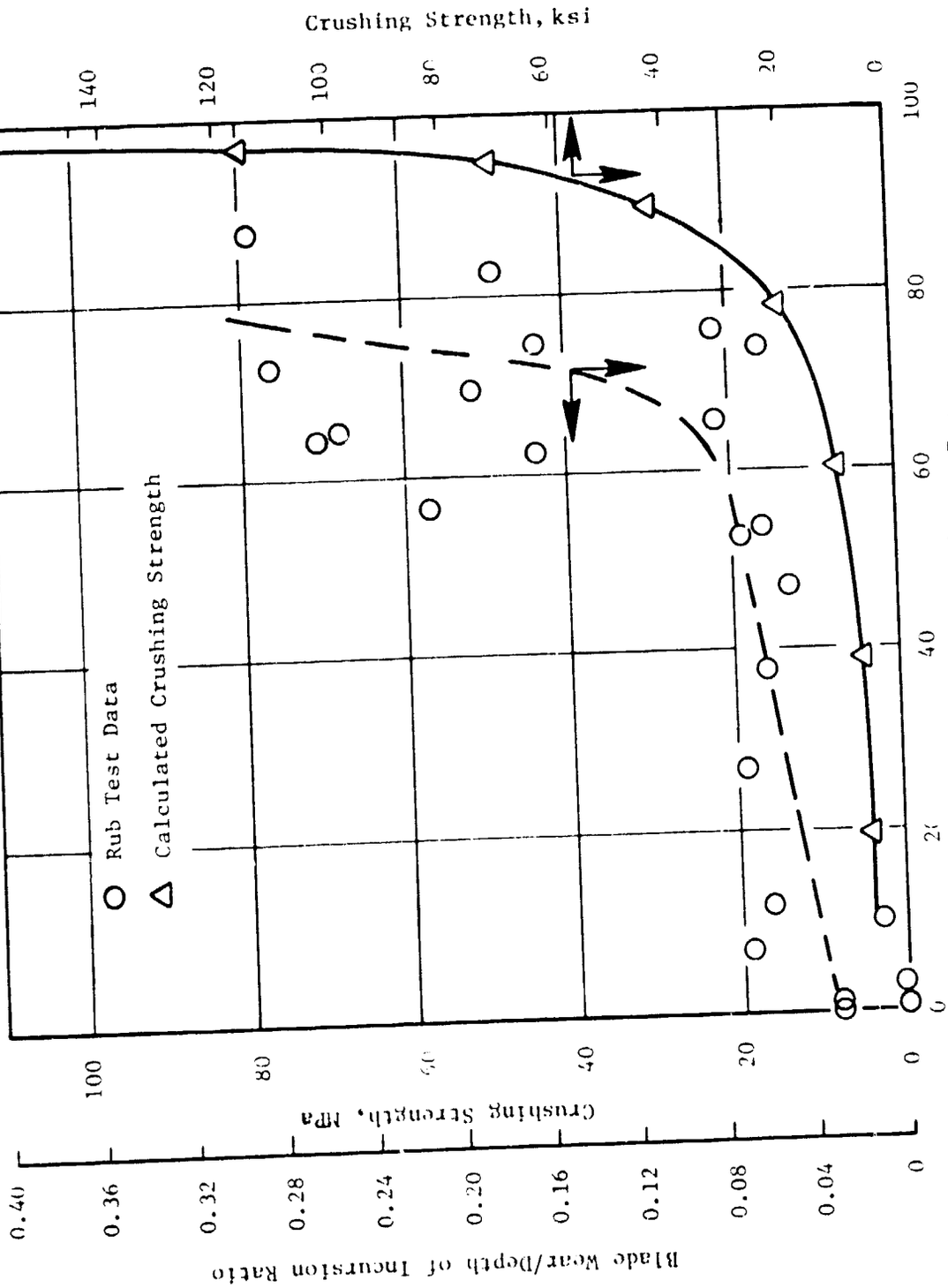


Figure 19. Relation Between Surface Hardness, Calculated Crushing Strength, and Blade Wear/Depth of Incursion Ratio for Ceramic Rub Layers Evaluated in This Project.

Figure 19 illustrates the relationship between surface hardness and crushing strength; harder layers have higher crushing strength as is generally expected. The abradability of a rub layer should depend on the crushing strength of the layer; layers with higher crushing strength should be more difficult to abrade (higher blade wear and tendency for blade material pick-up). The rub test data presented in Figure 19 show that the blade wear/depth of incursion ratio increases with increases in surface hardness of the layer and are in agreement with surface hardness/crushing strength/abradability relationship.

The surface hardness of the rub layers (Table 4) increased after the 1644 K (2500° F) 500-hour static oxidation exposure. This increase in hardness is believed to be due to the conversion of free silicon present in the layers to the cristobalite form of SiO_2. The hardness of Bradelloy 500 decreases considerably with oxidation exposure as shown in Table 4.

Cold Particle Erosion Test

The ceramic variations that were erosion tested in the as-fabricated condition (Table 5) showed low particle erosion resistance compared to Bradelloy 500. However, after a 1644 K (2500° F) 500-hour oxidation exposure Bradelloy 500 had poor erosion resistance, whereas the erosion resistance of the ceramic layers generally improved. The correlation between surface hardness of the layer and cold particle erosion resistance is shown in Figure 20. The data in this figure were obtained from layers evaluated in this program as well as from other in-house General Electric programs.

Ballistic Impact Test

Ballistic impact tests were made on specimens of all the variations in the statically oxidized condition (1644 K [2500° F] 500-hour exposure). The visual and fluorescent penetrant examination of the specimens indicated no cracks in the substrate blocks. However, the microstructural examination of the substrate block cut through the impact crater showed microcracks either in the substrate/layer interface or in the substrate block in some of the variations. The observations (Table 6) indicate that variations with thicker layers (Nos. 2, 3, 17, and 18) and higher additive levels in the layers (Nos. 5, 21, 22, 27, and 28) can withstand ballistic impact energy of 6.8 J (5 ft-lbf) without substrate microcracking. Also, the variation with the 1976 state-of-the-art (SOA) layer with SOA additive level on sintered SiC block (variation 26), and variations with SOA composition layers with SiC derived from graphite fiber on sintered SiC substrate block (Nos. 23 and 25) showed the absence of microcracks after the ballistic test. Ballistic impact tests on an oxidized Bradelloy 500 specimen caused multiple fractures in the specimen.

Table 5. Room Temperature Erosion Test Data on BN-Containing Material Systems.

Specimen Variation No.	Erosion Test Data - As Processed				Erosion Test Data After 1644 K (2500° F) 500-Hr Static Oxidation Exposure			
	Weight Loss, grams	Depth of Erosion, m x 10^{-3}	Erosivity Factor s/m x 10^4	(sec/mil)	Weight Loss, grams	Depth of Erosion, m x 10^4	Erosivity Factor s/m x 10^4	(sec/mil)
Bradelloy 500	0.035	0.2	21.7	(5.5)	0.278	1.7	2.8	(0.7)
1	0.044	0.7	5.3	(1.6)	0.032	0.3	13.0	(3.3)
2	0.046	0.6	7.9	(2.0)	0.047	0.4	9.8	(2.5)
3	0.039	0.4	11.0	(2.8)	0.037	0.5	8.3	(2.1)
4	0.043	0.3	25.0	(3.8)	0.034	0.4	13.0	(3.2)
5	0.188	2.2	2.0	(0.5)	0.063	0.8	5.5	(1.4)
6	0.116	1.7	2.8	(0.7)	0.083	0.2	4.7	(1.2)
7	0.095	1.2	3.9	(1.0)	0.094	0.8	5.5	(1.4)
8	0.074	0.8	5.5	(1.4)	0.057	0.7	6.3	(1.6)
9	0.111	1.2	3.9	(1.0)	0.057	1.1	3.9	(1.0)
10	0.311	2.4	2.0	(0.5)	0.110	1.1	4.3	(1.1)
11	0.365	2.4	2.0	(0.5)	0.110	1.1	3.9	(1.0)
12	0.566	2.2	2.0	(0.5)	0.290	2.1	2.0	(0.5)
13	0.031	0.3	17.0	(4.2)	0.039	0.6	7.5	(1.9)
14	0.037	0.4	13.0	(3.4)	0.038	0.6	7.5	(1.9)
15	0.027	0.3	17.0	(4.2)	0.048	0.4	10.0	(2.6)
16	0.098	2.1	2.0	(0.5)	0.053	0.5	9.4	(2.4)
17	0.028	0.4	13.0	(3.2)	0.032	0.3	15.0	(3.9)
18	0.031	0.4	11.0	(2.7)	0.039	0.4	12.0	(3.0)
19	0.049	1.2	3.9	(1.0)	0.052	0.6	7.5	(1.9)
20	0.048	0.8	5.9	(1.5)	0.057	0.6	7.5	(1.9)
21	0.071	1.0	4.3	(1.1)	0.043	0.5	9.4	(2.4)
22	0.087	1.7	2.8	(0.7)	0.061	0.8	5.9	(1.5)
23	0.071	1.0	4.3	(1.1)	0.071	0.7	6.7	(1.7)
24	0.064	0.8	5.5	(1.4)	0.042	0.3	17.0	(4.2)
25	0.049	0.6	7.9	(2.0)	0.051	0.5	9.8	(2.5)
26	0.028	0.4	11.0	(2.8)	0.032	0.5	9.4	(2.4)
27	0.089	0.9	4.7	(1.2)	0.043	0.4	10.0	(2.6)
28	0.129	2.0	2.4	(0.6)	0.036	0.3	14.0	(3.5)

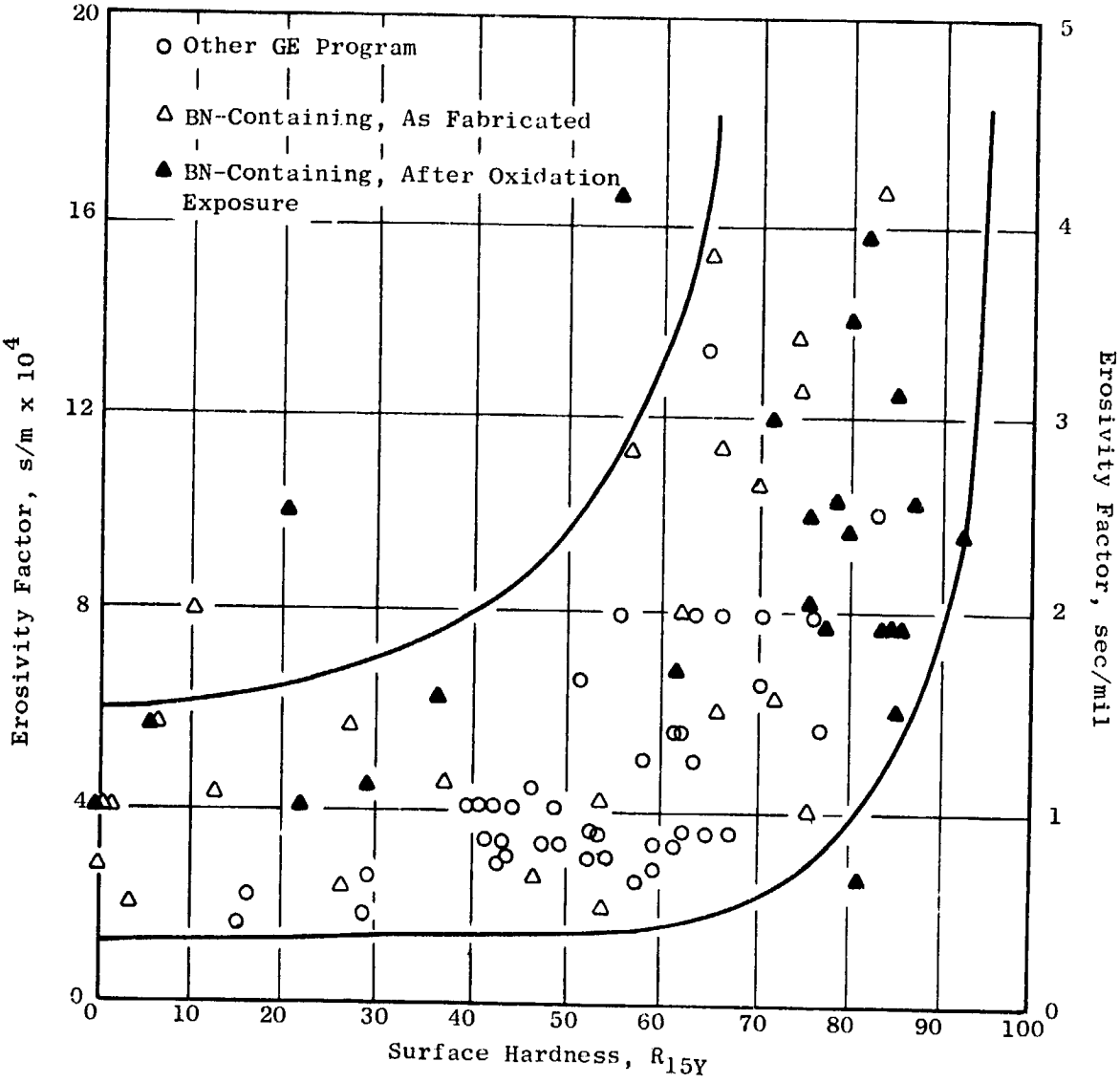


Figure 20. Erosivity Factor Versus Hardness of BN-Containing Rub Layers.

Table 6. Ballistic Impact Test Results on BN Containing Material Systems.

Specimen Variation No.	Depth of Impact $m \times 10^{-3}$	(mils)	Microcracks in Substrate or Substrate/Layer Interface Near the Impact Crater
Bradelloy 500	---	---	Yes
1	1.14	(45.0)	Yes
2	1.10	(43.5)	No
3	0.95	(38.0)	No
4	1.20	(47.3)	NE
5	1.61	(63.2)	No
6	1.74	(68.5)	Yes
7	1.35	(53.2)	Yes
8	1.07	(42.3)	Yes
9	1.67	(65.8)	Yes
10	1.57	(62.0)	Yes
11	1.83	(72.0)	Yes
12	2.54	(100.0)	Yes
13	1.31	(51.5)	Yes
14	1.07	(42.0)	Yes
15	0.83	(32.6)	Yes
16	2.00	(78.7)	Yes
17	1.34	(52.7)	No
18	7.70	(30.3)	No
19	1.36	(53.4)	NE
20	0.98	(38.7)	NE
21	1.01	(39.7)	No
22	1.32	(51.9)	No
23	1.68	(66.0)	No
24	1.19	(47.0)	Yes
25	1.28	(50.5)	No
26	0.81	(32.0)	No
27	1.16	(45.5)	No
28	1.22	(48.2)	No

NE = Not examined, specimen used in other tests

Selection of the Three Most Promising Candidates

The data from all the initial screening tests were analyzed to select the three most promising variations for the advanced screening tests in Task I. The variations were ranked according to their cold particle erosion resistance in the as-fabricated condition and their oxidation resistance (Table 7).

The following two selection requirements were applied to the 10 best candidates based on cold particle erosion resistance (Table 8).

1. No scabbing in room temperature abradability tests.

2. No gross cracking and no microcracking in the substrate block in ballistic impact tests.

Based on these requirements, Variations 15, 13, and 14 (ranked 1, 2, and 4) were rejected. Variation 4 was concluded to be nonrepresentative of the SOA + 5 g BN composition based on microstructural examinations and properties comparisons with other SOA + 5 g BN composition layers, and was not considered further for selection. Variations 17, 3, 26, 18, and 2 (ranked 5 through 9) had surface layers consisting essentially of the SOA composition with SiC derived from graphite powder (Figure 1). The surface layer thickness/substrate thickness (SLT/ST) was the only other variation in these selections. The ballistic impact test on Variation 26 indicated that the surface layer with a standard SLT/ST ratio was able to withstand 6.8 J (5 ft-lbf) impact energy without microcracking in the substrate block and it was not necessary to use layers with higher SLT/ST ratio for withstanding ballistic impact energy of 6.8 J (5 ft-lbf). The thicker surface layers (SLT/ST = 0.17 and 0.42) had greater oxidation weight gains and are ranked 25 through 28 among the 28 variations (Table 7). Based on these data, the surface layer with the 1976 SOA composition with SiC derived from graphite powder was considered as the most promising variation. Variation 26, which has this surface layer on a sintered SiC substrate block, was selected as one of the variations to be evaluated in the advanced screening tests in Task I. As mentioned earlier, the cloth-based Silcomp^{TM} substrate blocks showed lower oxidation weight gains than the sintered SiC and tow-based Silcomp^{TM} blocks. In view of this, the 1976 SOA surface layer applied to a cloth-based Silcomp^{TM} block with high volume percent SiC (similar to Variation 6) was selected as the second variation for advanced screening tests.

The particle erosion resistance of the 1976 SOA layer (Variation 26) tested in the as-fabricated condition showed low values compared to Bradelloy 500. Further improvement in the cold particle erosion resistance was expected with a harder layer fabricated by using a lower level of BN additive (SOA - 5 g BN) relative to the 1976 SOA layer. This layer on a cloth-based Si/SiC substrate was selected as the third variation for advanced screening.

An independent analysis of the test data conducted by Mr. Stan Young of NASA-Lewis, based on equal weight to each test (cold particle erosion resistance before and after static oxidation, room temperature rub, static oxidation and ballistic impact), showed Variation 25 to have the best overall

Table 7. Ranking of BN-Containing Material Systems Based on Erosion and Oxidation.

Rank	Variation No., Based on Cold Particle Erosion Resistance in the As-Processed Condition	Variation No., Based on Weight Gain After 1644 K (2500° F) 500-Hour Oxidation
1	15	6
2	13	1
3	4	5
4	14	9
5	17	16
6	3	21
7	26	20
8	18	10
9	2	7
10	25	11
11	1	13
12	20	12
13	8	4
14	24	14
15	27	15
16	21	24
17	23	25
18	19	22
19	7	23
20	19	8
21	22	28
22	6	19
23	28	27
24	5	26
25	10	17
26	11	2
27	12	18
28	16	3

Table 8. Combined Assessment of Erosion Resistance, Rub Test Scabbing, and Ballistic Impact Cracking in BN-Containing Material Systems.

Rank in Cold Erosion Test	Specimen	Scab	Ballistic Impact Microcrack
1	15	No	Yes
2	13	Yes	Yes
3	4	No	Not Examined
4	14	No	Yes
5	17	No	No
6	3	No	No
7	26	No	No
8	18	No	No
9	2	No	No
10	25	No	No
	Bradelloy 500	Yes	Yes

ranking. The GE ranking for Variation 25 was sixth. This variation did not follow the property trend shown by similar layers with SiC derived from graphite fiber which had shown poor erosion resistance. To examine a possibility of a synergistic effect of the higher BN level and the graphite fiber, it was agreed to add Variation 25 to the above three variations (Table 9) for further evaluation in Task I Advanced Screening.

4.2 TASK I - ADVANCED SCREENING

Hot Rub Tests

The results of the rub tests on the four variations listed in Table 9 are shown in Table 10. Figures 21 through 23 show the rubbed surfaces for each variation. The important observations of the rub tests were the following:

1. All the specimens rubbed well. There were some isolated spots which showed metal pickup. Based on the blade wear/depth of incursion ratio and tendency for metal pickup, Variation 4 was ranked best followed by Variations 2, 3, and 4.

2. The sintered silicon carbide substrate block in Variation 1 fractured into two pieces as shown in Figure 21(a). This specimen also exhibited the highest amounts of blade material transfer and blade wear ratio. Rubs in the remaining specimens did not cause fracture of the substrate blocks.

3. The shear force in all specimens during the rub was less than 2.2 N (0.5 lb).

Mach 1.0 Oxidation/Erosion Test

The first attempt to run the Mach 1.0 oxidation/erosion test at 1589 K (2400° F) resulted in fracture of all the specimens near the base where they were being held in the cooled metal holder. Therefore, the second test was run with a modified holder on new specimens at 1477 K (2200° F) for 84 hours. The test was run at 1589 K (2400° F) for 16 hours unintentionally due to clogging of the IRCON window during the overnight run after the specimens had completed 15 hours at 1477 K (2200° F). Figure 6 shows the Mach 1.0 oxidation/erosion test specimens assembled in the holder, and Figure 24 shows the typical geometry of the specimen. Figures 25 and 26 show the test specimens after 100 hours of exposure time. All the specimens except Specimen 4 completed the 100 hour test. Figures 27 through 29 show the specimens after 31, 45, 67, and 100 hours of exposure. Specimen 4 (SOA + 10 g BN composition layer with SiC derived from graphite fiber on a sintered SiC substrate block) fractured at the base where it was being held in the metal holder after one cycle to 1477 K (2200° F) (Figure 26). The layer on this specimen showed signs of local gas

Table 9. BN-Containing Material System Variations for Task I Advanced Screening Tests.

	Substrate	Surface Layer	Substrate Volume % SiC	Additive Level	SLT/ST
1.	Sintered dense SiC	1976 SOA composition (BN) with SiC derived from graphite powder	---	SOA	S
2.	Cloth-based Si/SiC	1976 SOA composition (BN) with SiC derived from graphite powder	High	SOA	S
3.	Cloth-based Si/SiC	1976 SOA composition (BN) with SiC derived from graphite powder	High	SOA - 5 g BN	S
4.	Sintered dense SiC	SOA compositon (BN) with SiC derived from graphite fiber	---	SOA + 10 g BN	S

SLT/ST = surface layer thickness/substrate thickness ratio

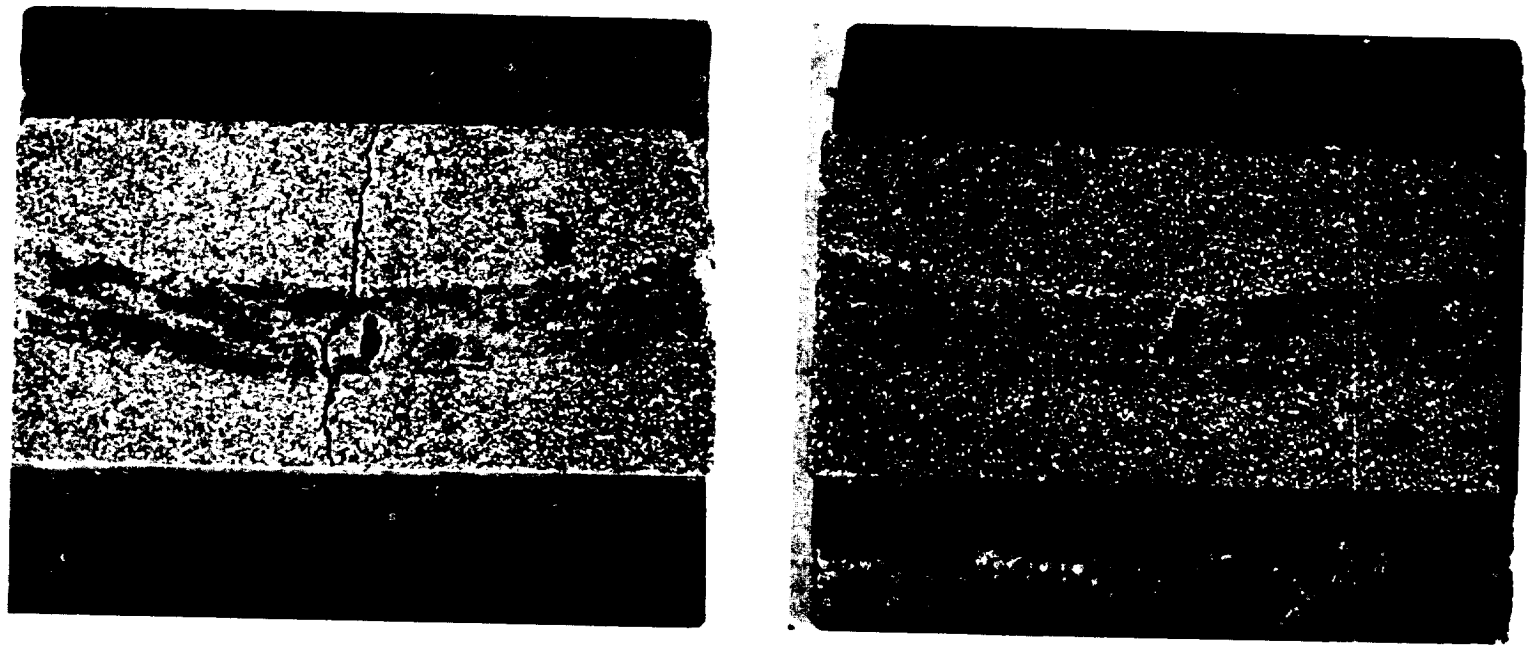
SOA = State of the art

S = Standard SLT/ST

Table 10. Hot Rub Test Data on BN-Containing Material Systems.

Specimen Variation No. (Table 9)	Surface Hardness (R_{15Y})	Test Temp K	Test Temp (° F)	Shear Force N	Shear Force (lb)	Depth of Rub m x 10^{-3}	Depth of Rub (mils)	Average Blade Wear m x 10^{-3}	Average Blade Wear (mils)	Scab	Depth of Incursion m x 10^{-3}	Depth of Incursion (mils)	Blade Wear/ Depth of Incursion	Remarks	Rank (Best Rub Given, Rank 1)
1	67	933	(1220)	<2.2	(<½)	0.25	(10)	0.445	(17.5)	Yes, spotty over most of the rub path	0.69	(27)	0.65	Fracture of substrate	4
2	48	883	(1130)	<2.2	(<½)	0.6	(24)	0.432	(17)	Yes, in isolated areas	1.00	(41)	0.41		2
3	67	955	(1260)	<2.2	(<½)	0.58	(23)	0.432	(17)	Yes, 1/3 of the specimen	1.00	(40)	0.42		3
4	Soft*	877	(1120)	<2.2	(<½)	0.65	(26)	0.102	(4)	No	0.76	(30)	0.14		1

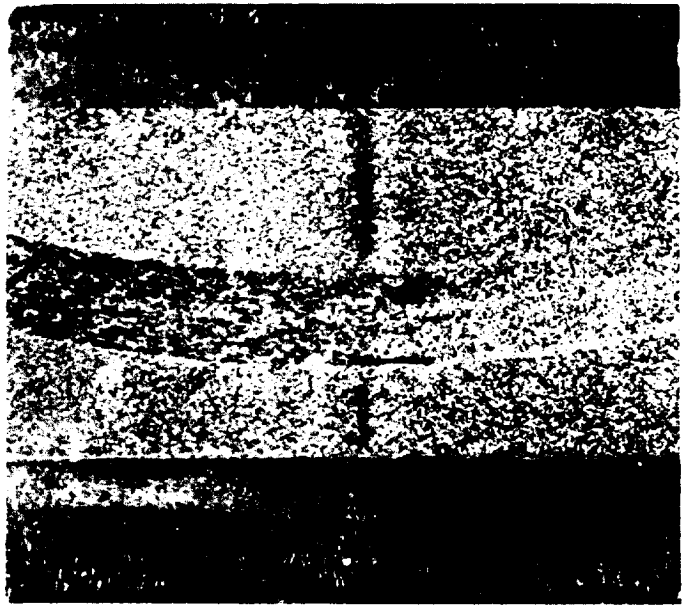
* The original specimen evaluated in preliminary screening tests (Variation 25) had an R_{15Y} hardness of 10 which increased to 20 after 1644° K, 500 hours of static oxidation exposure.



(a) Variation 1 2X

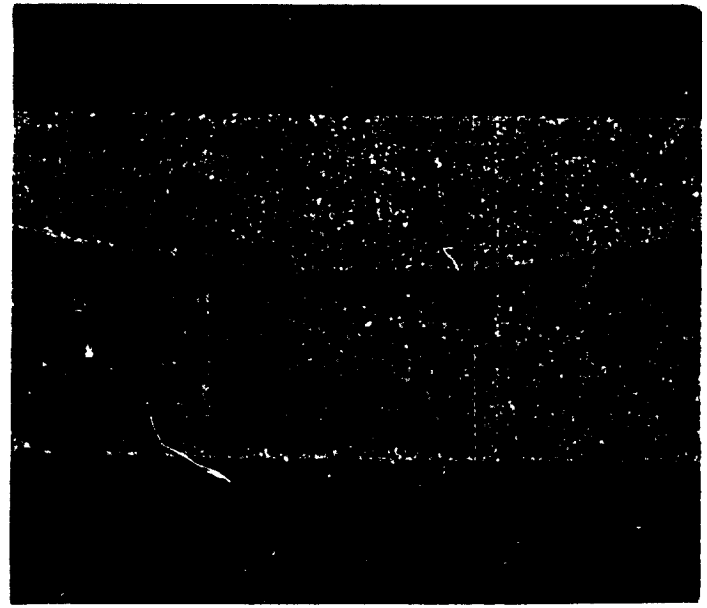
(b) Variation 2 2X

Figure 21. Rub Surface of Task I Advanced Screening Variations 1 and 2 After Hot Abradability Test.



2X

Figure 22. Rub Surface of Task I Advanced Screening Variation 3 After Hot Abradability Test.



2X

Figure 23. Rub Surface of Task I Advanced Screening Variation 4 After Hot Abradability Test.

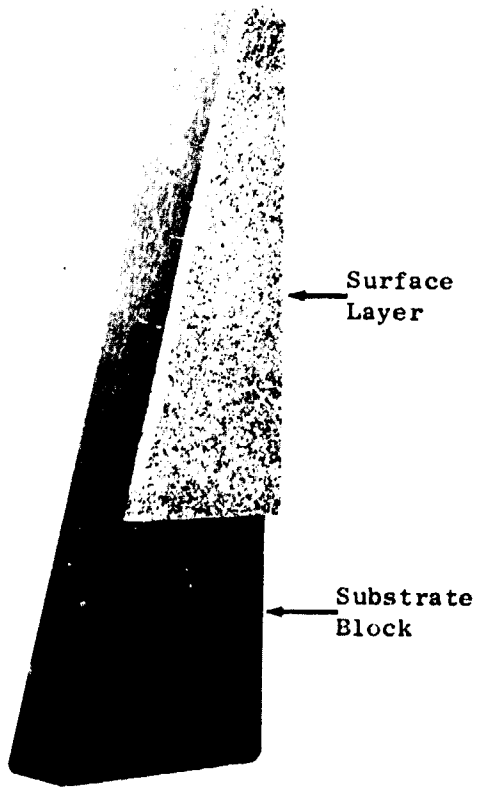
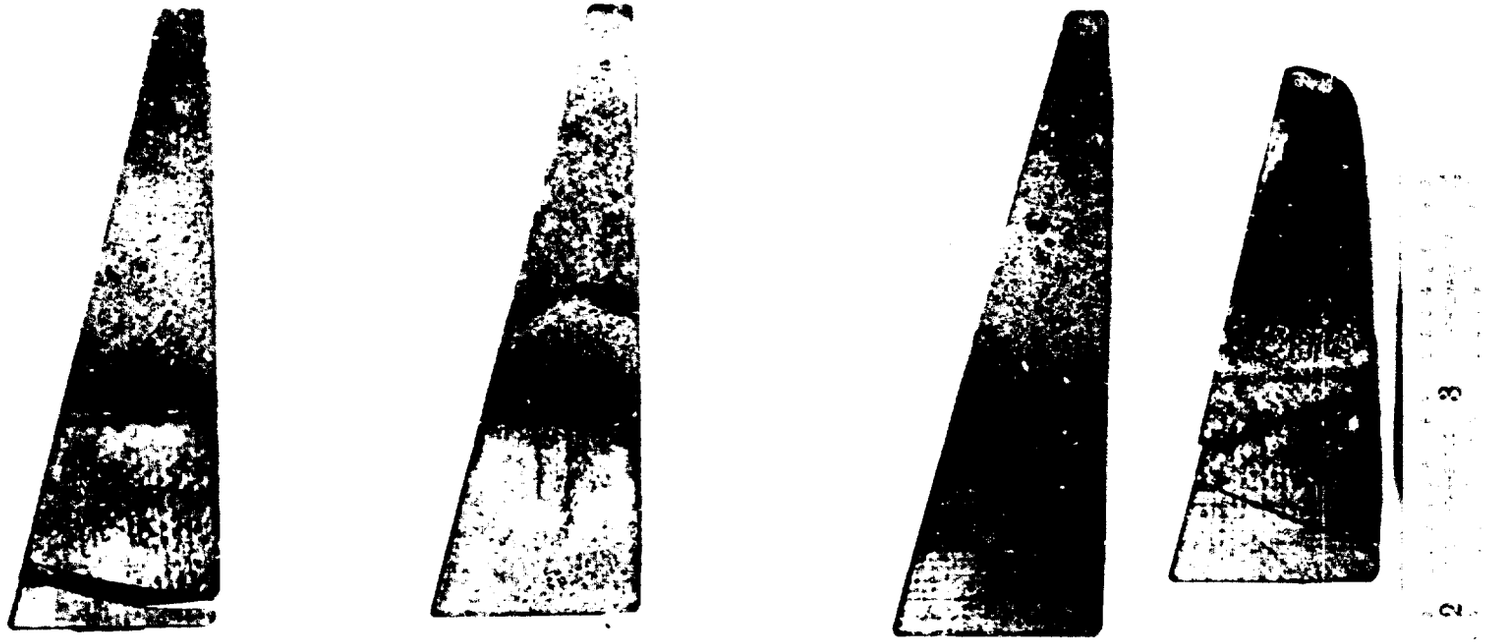


Figure 24. Mach 1.0 Gas Oxidation/Erosion Test Specimen Configuration.

Figure 25. Task I Mach 1.0 Oxidation Test Specimens After 84 Hours at 1477 K (2200° F) and 16 Hours at 1589 K (2400° F).



Specimen 1 Specimen 2 Specimen 3 Specimen 4

Figure 26. Task I Mach 1.0 Oxidation Test Specimens Showing the Rub Layers After 84 Hours of Exposure at 1477 K (2200° F) and 16 Hours at 1589 K (2400° F).

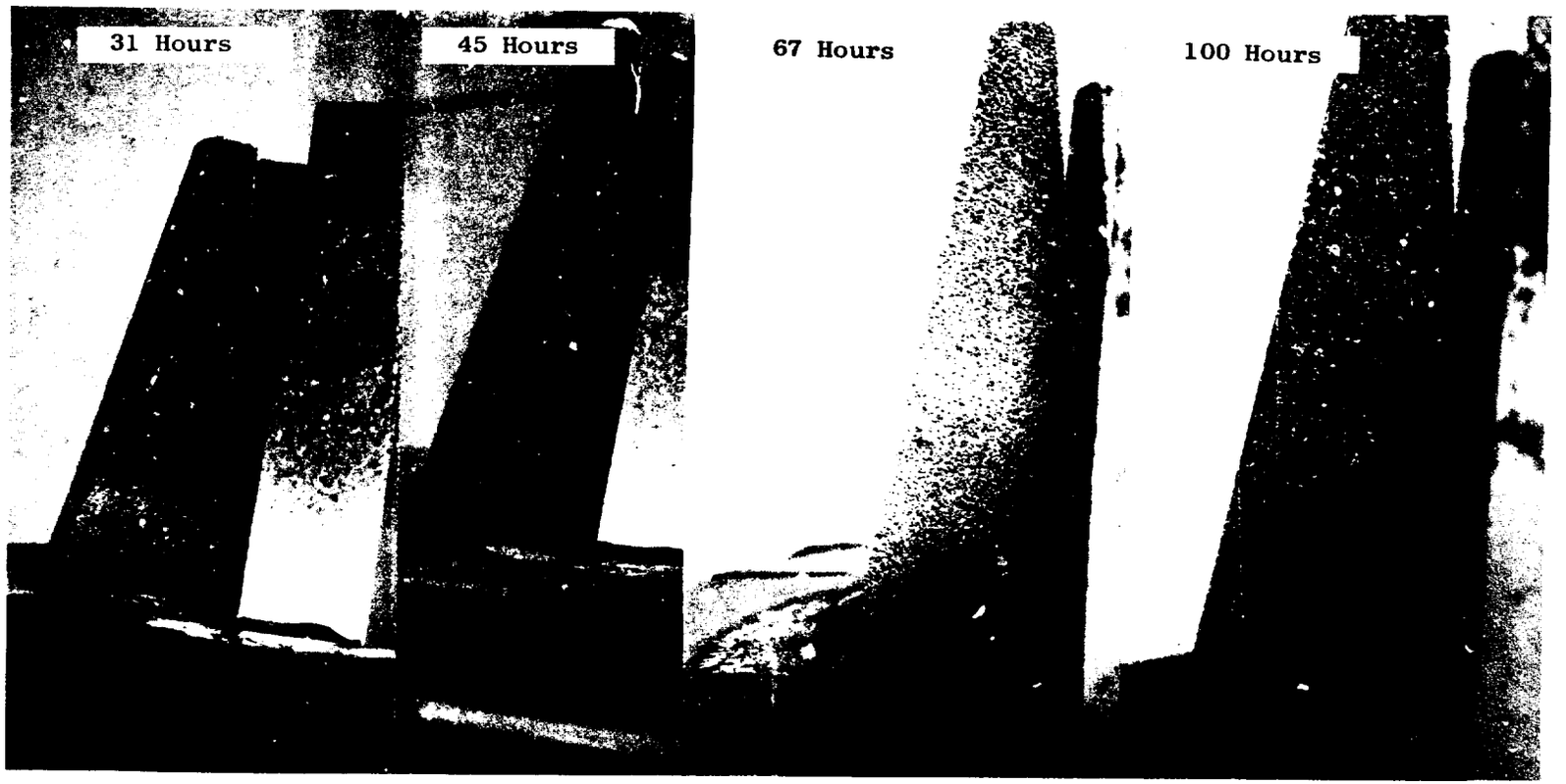


Figure 27. Task I Mach 1.0 Oxidation Test Specimen 1 After 31, 45, 67, and 100 Hours of Exposure.

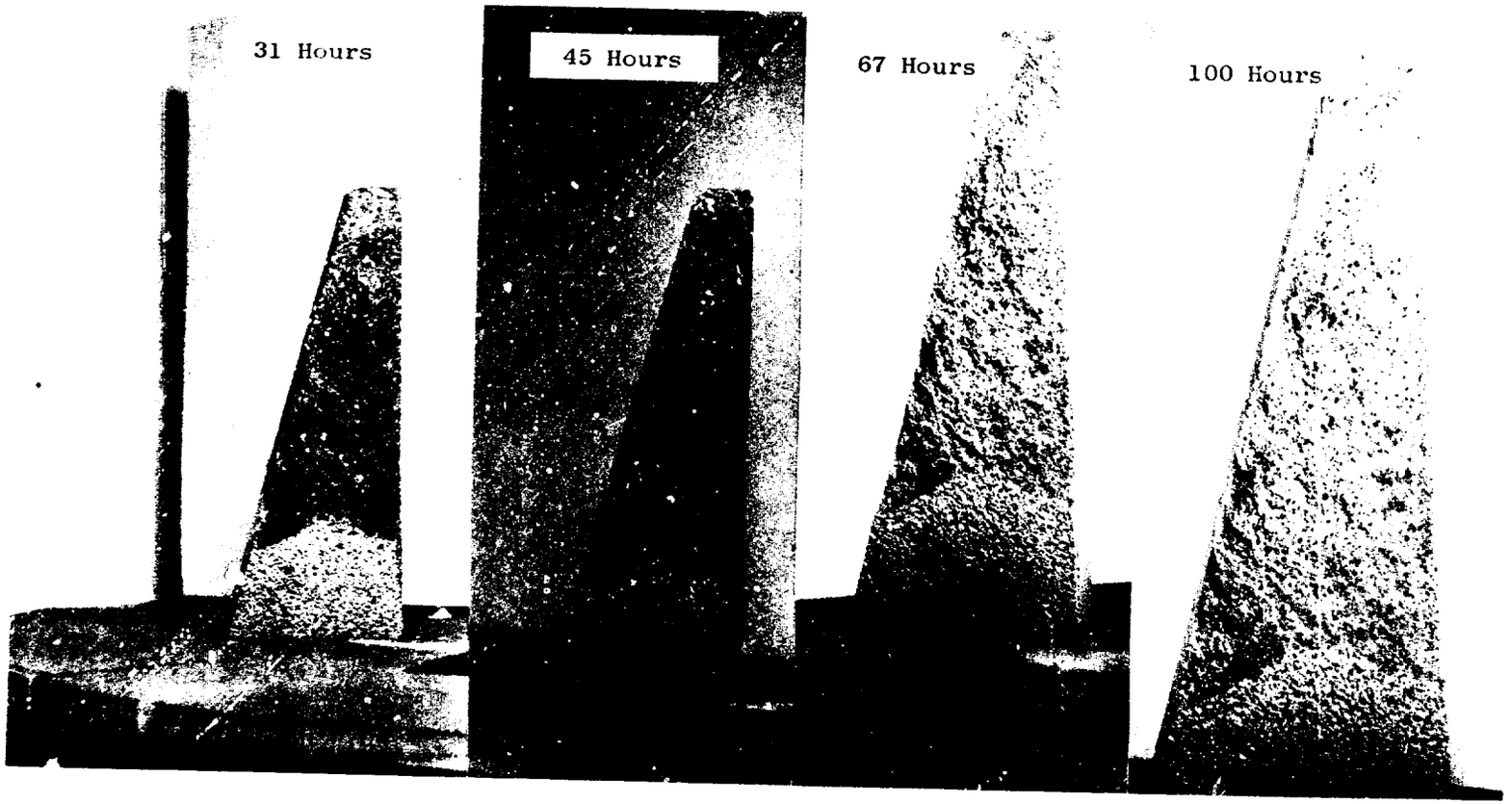


Figure 28. Task I Mach 1.0 Oxidation Test Specimen 2 After 31, 45, 67, and 100 Hours of Exposure.

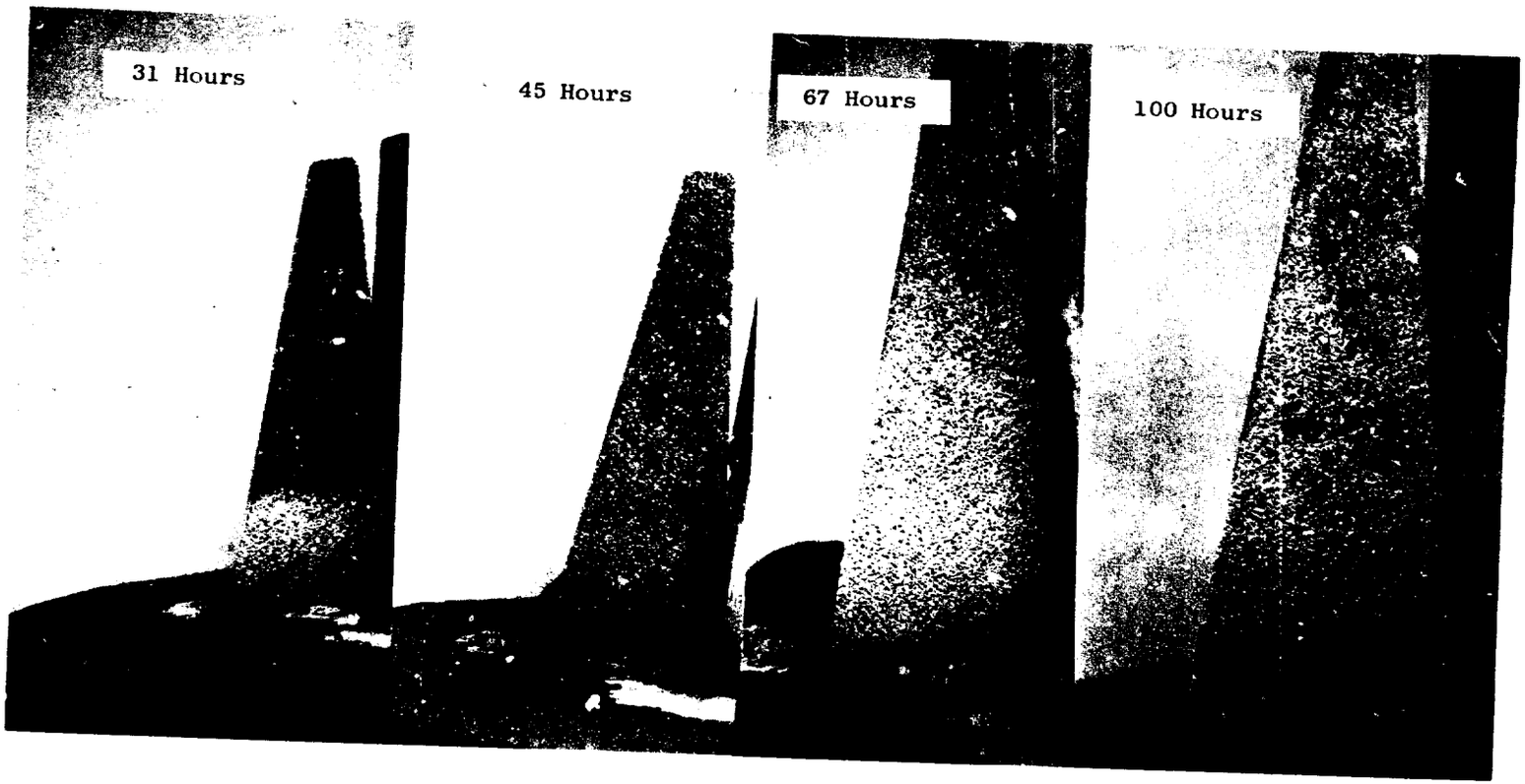


Figure 29. Task I Mach 1.0 Oxidation Test Specimen 3 After 31, 45, 67, and 100 Hours of Exposure.

erosion. In the first Mach 1.0 gas oxidation/erosion test the same layer had shown considerable gas erosion at the tip after only one cycle at 1589 K (2400° F).

The weight, thickness, and surface hardness measurements were taken before and after the test. After the completion of the test, the specimens were evaluated for room temperature abradability and cold particle erosion resistance. The results are summarized in Table 11 from which the following observations were made:

1. Material systems of Specimens 1 and 3 survived Mach 1.0 gas oxidation/erosion and thermal shock at 1589 K (2400° F) for at least 16 hours with no signs of layer erosion. The layer in Specimen 2 eroded excessively during overtemperature to 1589 K (2400° F) in the early part of the test. No further layer degradation was observed in this specimen when the test was continued at 1477° F (2200° F).

   Specimens 1 and 3 showed an average increase in thickness of about 1.27×10^{-5} m to 3.81×10^{-5} m (0.05-1.5 mils) and a weight loss of about 1%: specimen thickness = 1.27×10^{-5} m (0.5 inch).

   During the oxidation exposure, the BN particles oxidized away leaving holes where the BN particles had been. This effect made the structure of the layer essentially a lacy network of Si + SiC + SiO_2 (Figures 30 and 31) through the total thickness of the layer.

2. Cloth-based Si/SiC (Silcomp^{TM}) as substrate block survived Mach 1.0 gas oxidation/erosion and thermal shock up to 1589 K (2400° F). The sintered silicon carbide was more fracture-sensitive compared to Silcomp^{TM}. However, in all cases the fractures occurred in the area where the specimens were being held in the metal holder slots. No fracture or crack was observed in the specimen area exposed to the Mach 1.0 gas stream.

3. A correlation between the layer surface hardness and hot gas erosion resistance was found. No correlation between the cold particle erosion resistance and hot gas erosion resistance was observed.

4. No significant change in cold particle erosion resistance of the layer was observed after the Mach 1.0 gas oxidation exposure.

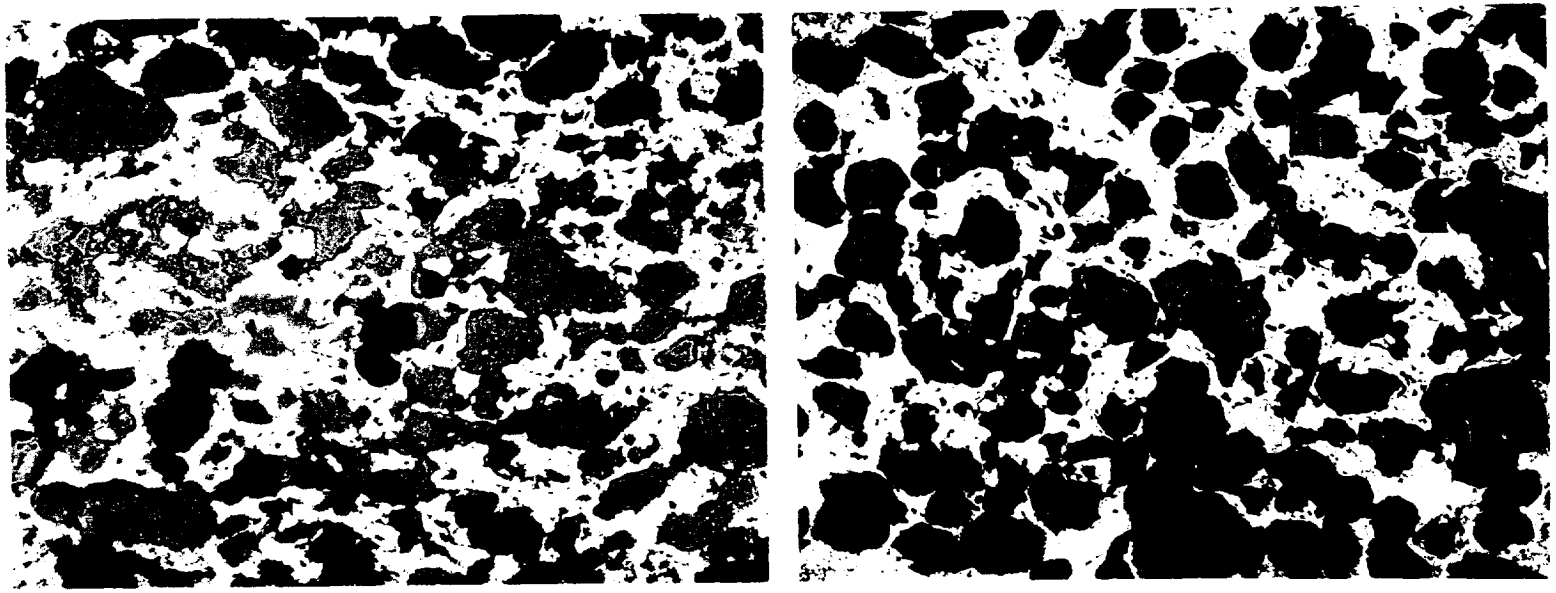
5. The Mach 1.0 gas oxidized layers (Specimens 1, 2, and 4, R_{15Y} = 55, 21, and 18, respectively) rubbed with acceptable blade wear and no metal transfer (scabbing). The layer with surface hardness (R_{15Y}) of 91 (Specimen 3) caused excessive blade wear and scabbing.

Table 11. Task I Mach 1.0 Oxidation/Erosion Test Results.

Specimen	Layer Surface Hardness Before Exposure, R_{15Y}	Layer Surface Hardness After Exposure, R_{15Y}	Layer Erosion	Substrate Cracking	Blade Wear/ Depth of Incursion	Scab	Erosivity Factor in Cold Particle Erosion Test	
							s/m X 10^4	(sec/mil)
BN + Si/SiC Layer on Sintered SiC Block	53	55	No	Yes, One Bottom Edge	0.28	No	2.5	(2.4)
BN + Si/SiC Layer on Si/SiC Block	46	25 (on Eroded Areas)	Badly Eroded at 2400° F	No	0.21	No	9.5	(2.4)
BN + Si/SiC Layer on Si/SiC Block	93	91	No	No	0.53	Yes	5.1	(1.3)
BN + Si/SiC Layer on Sintered SiC Block	Very Soft, Goes Beyond Scale	18	Yes, in One Cycle to 2200° F	Yes	0	No	3.9	(1.0)

Figure 30. Lacy Si + SiC + SiO_2 Structure of BN-Containing Layer After Mach 1.0 Cyclic Oxidation Exposure.

- The black and gray areas are voids where BN grains vaporized; the voids are surrounded by a network of Si + SiC + SiO_2 (50X).



Specimen 1 (Table 11) 50X

Specimen 3 (Table 11) 50X

Figure 31. Typical Microstructures of Cross Sections of Rub Layers After Mach 1.0 Oxidation/Erosion Test.

Based on the Mach 1.0 oxidation/erosion and hot rub tests, the surface layer of 1976 SOA Composition (BN) with SiC derived from graphite powder and with the surface hardness R_{15Y} of 60 ± 10 showed the most potential for thermal shock, oxidation, and hot gas erosion resistances and for nonscabbing behavior in rub tests. This conclusion was in agreement with observations derived from other GE programs evaluating similar surface layers.

Therefore, Task III Advanced Laboratory Evaluations were made on the surface layers of 1976 SOA composition (BN) with SiC derived from graphite powder and with surface hardness (R_{15Y}) of 60 ± 10 on both Silcomp™ and hot pressed silicon carbide and substrate blocks.

Based on the preliminary screening tests, Silcomp™ emerged as the most promising substrate block material. Since hot pressed silicon carbide block seals with BN-containing layers were being evaluated under the NASA-sponsored Energy Efficient Engine (E^3) program (NAS3-20643) along with Silcomp™ in Mach 1.0 gas oxidation/erosion tests in Task III. The Mach 1.0 gas oxidation/erosion test data from this program formed one of the criteria for selection of an approach for ceramic shroud development in the E^3 program.

4.3 TASK II - EVALUATION OF BN-FREE MATERIAL SYSTEMS

The nine variations of density and pore size evaluated in BN-free materials are listed in Table 2. Two specimens for each variation were fabricated and after metallographic and density evaluations (Figures 32 and 33) the following tests were completed in the order mentioned:

1. Surface hardness measurement
2. Cold particle erosion test
3. Room temperature rub test
4. 1644 K (2500° F) static oxidation test
5. Surface hardness measurement
6. Cold particle erosion test
7. Ballistic impact test

Surface Hardness, Erosion Test, and Rub Test Results

The surface hardness, cold particle erosion test, and rub test data are shown in Table 12 and in Figures 34 through 37. The surface hardness (R_{15Y}) data were averages of nine readings and the cold particle erosion test data

- The black areas are pores, the light gray areas are SiC, the white areas are Si, and the dark gray areas inside the SiC network are unreacted carbon.

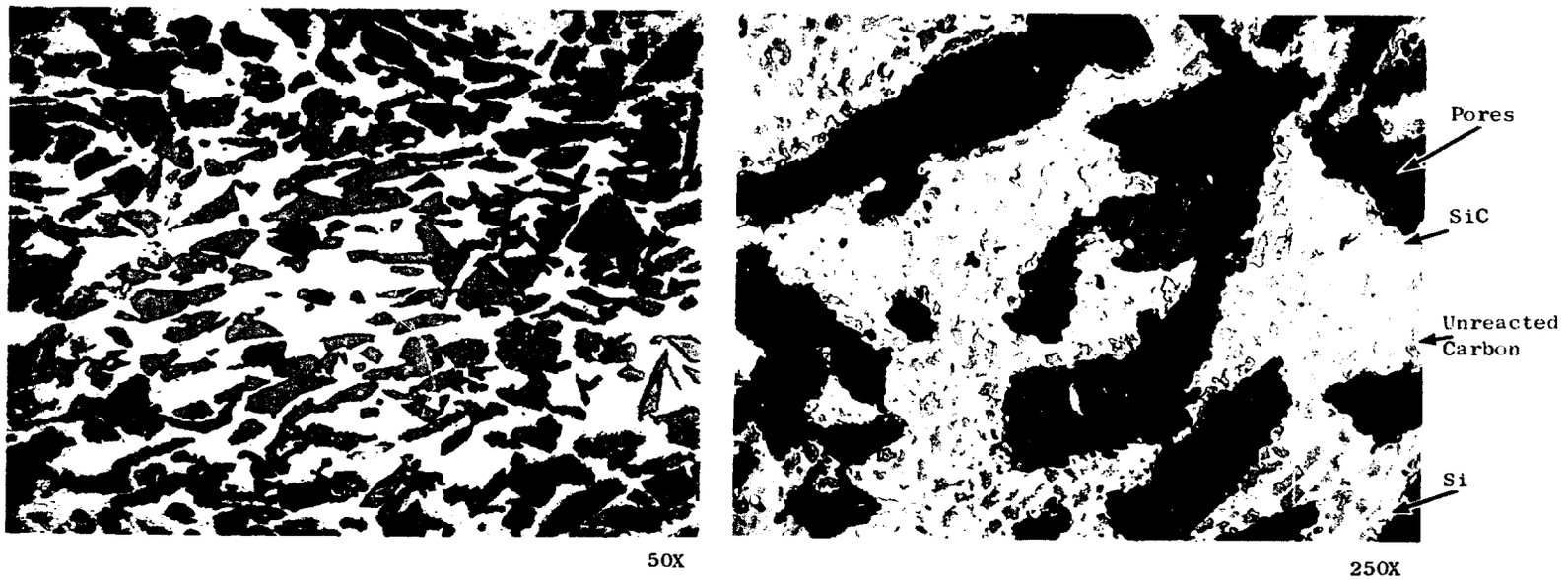
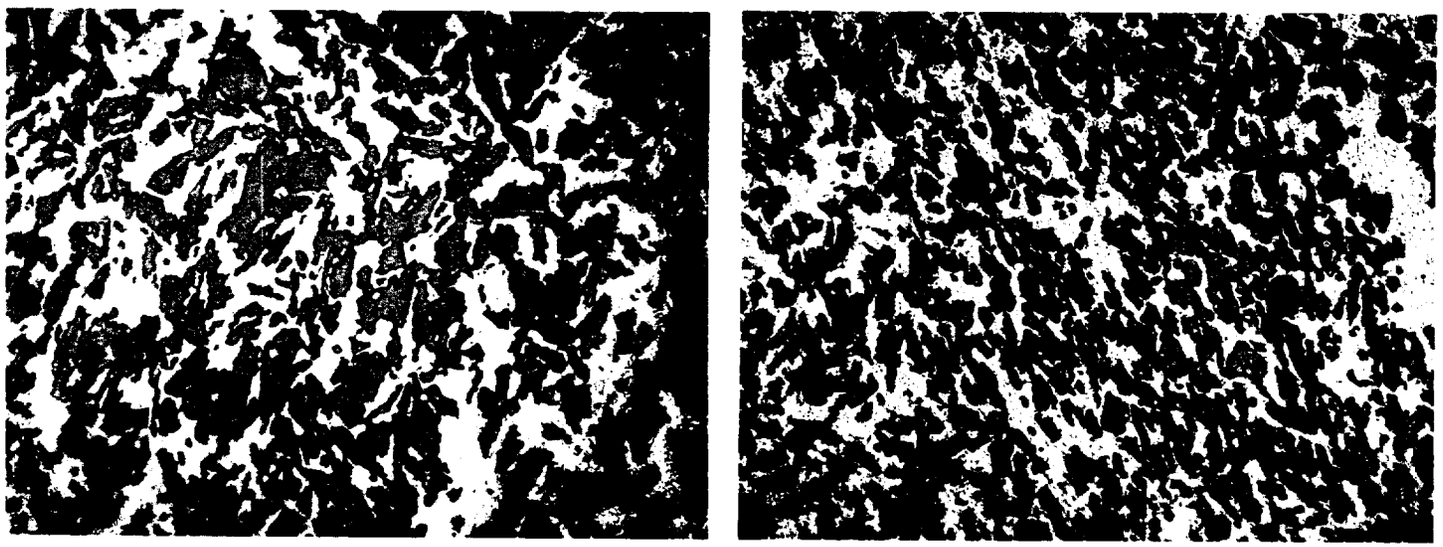


Figure 32. Typical Microstructure of a BN-Free Material of 1.45×10^3 kg/m^3 (1.45 g/cm^3) Density and 250 μm Pore Size.



(a) 180 μm Pore Size 50X (b) 125 μm Pore Size 50X

Figure 33. Typical Microstructure of a BN-Free Material of 1.45 x 10^3 kg/m^3 (1.45 g/cm^3) Density at 180 μm and 125 μm Pore Sizes.

Table 12. Preliminary Screening Tests Data on BN-Free Materials in the As-Fabricated Condition.

Specimen Variation No.	Density, $kg/m^3 \times 10^3$	Hardness R_{15Y}	Depth of Erosion		Weight Loss,	Erosivity Factor,		Blade Wear,		Blade Wear/ Depth of Incursion	Scab
			$m \times 10^4$	(mil)	$kg \times 10^{-6}$	$s/m \times 10^{-5}$	(sec/mil)	$m \times 10^{-5}$	(mil)		
1	1.26	39	5.46	(21.5)	38.6	0.87	(2.1)	0	(0)	0	No
1	1.24	21	5.78	(21.2)	38.7	0.84	(2.1)	2.54	(1)	0.06	No
2	1.24	25	3.02	(11.9)	23.2	1.50	(3.8)	2.54	(1)	0.06	No
2	1.23	7	4.37	(17.2)	29.0	1.01	(2.6)	0	(0)	0	No
3	1.26	18	3.68	(14.5)	22.6	1.21	(3.1)	5.08	(2)	0.13	No
3	1.30	39	2.64	(10.4)	15.3	1.71	(4.3)	5.08	(2)	0.14	No
4	1.32	56	3.35	(13.2)	28.0	1.3[illegible]	(3.4)	5.08	(2)	0.13	No
4	1.34	54	3.12	(12.3)	28.6	1.42	(3.6)	2.54	(1)	0.07	No
5	1.34	54	3.43	(13.5)	20.2	1.30	(3.3)	2.03	(0.8)	0.05	No
5	1.34	51	3.12	(12.3)	18.8	1.45	(3.7)	1.52	(0.6)	0.04	No
6	1.37	48	1.96	(7.7)	10.2	2.31	(5.8)	5.08	(2.0)	0.13	No
6	1.32	33	3.05	(12.0)	15.0	1.50	(3.7)	2.54	(1.0)	0.07	No
7	1.46	68	0.69	(2.7)	10.2	6.51	(16.6)	2.54	(1.0)	0.06	No
7	1.44	61	0.97	(3.8)	12.7	4.61	(11.8)	5.08	(2.0)	0.13	No
8	1.46	67	0.81	(3.2)	6.8	5.50	(14.1)	2.54	(1.0)	0.07	No
8	1.43	64	0.53	(2.1)	6.8	8.40	(21.4)	2.54	(1.0)	0.07	No
9	1.50	66	0.78	(1.5)	3.7	12.1	(30.0)	13.5	(5.3)	0.33	No
9	1.47	59	0.41	(1.6)	5.0	11.1	(28.1)	7.62	(3.0)	0.19	No

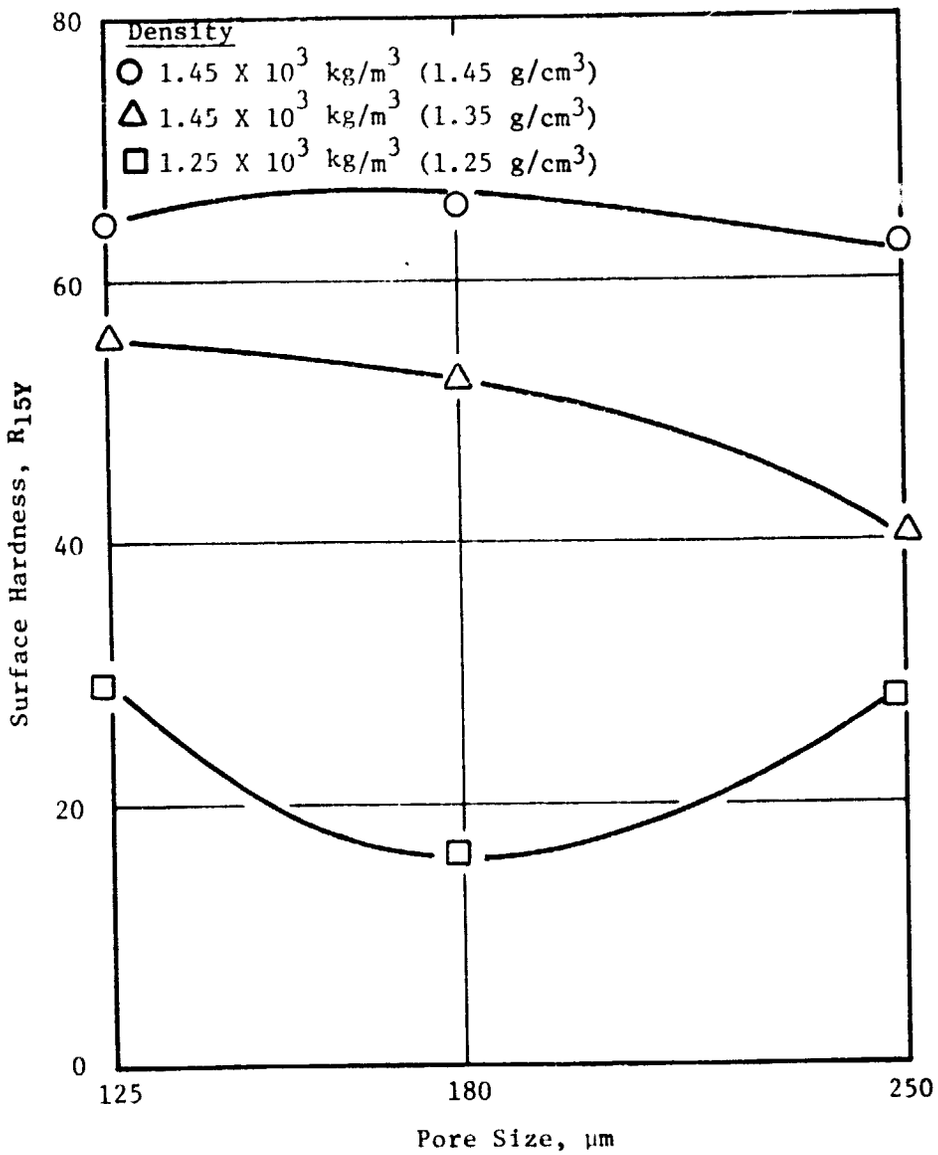


Figure 34. Effect of Pore Size on Surface Hardness in BN-Free Materials.

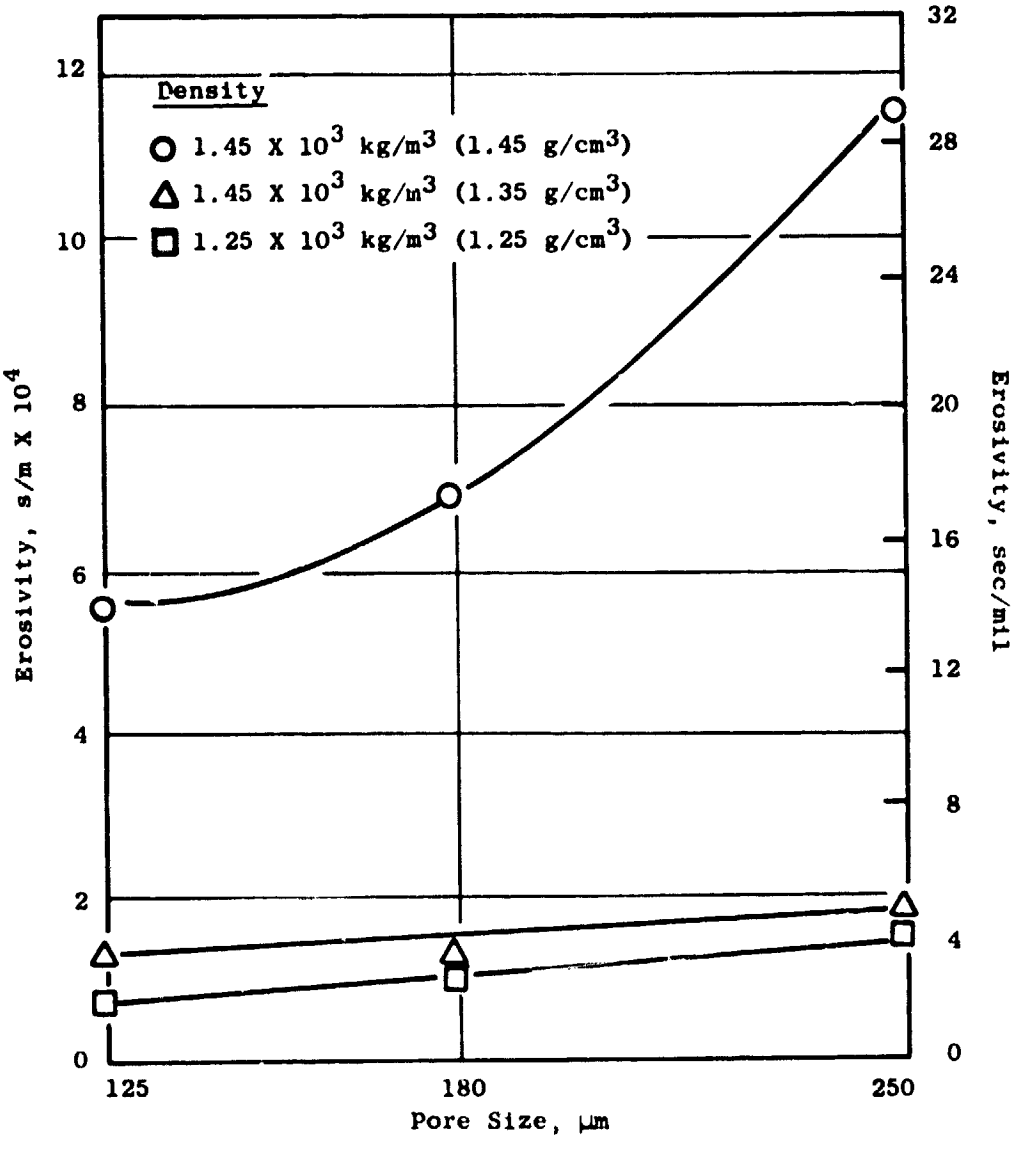


Figure 35. Effect of Pore Size on Erosivity in BN-Free Materials.

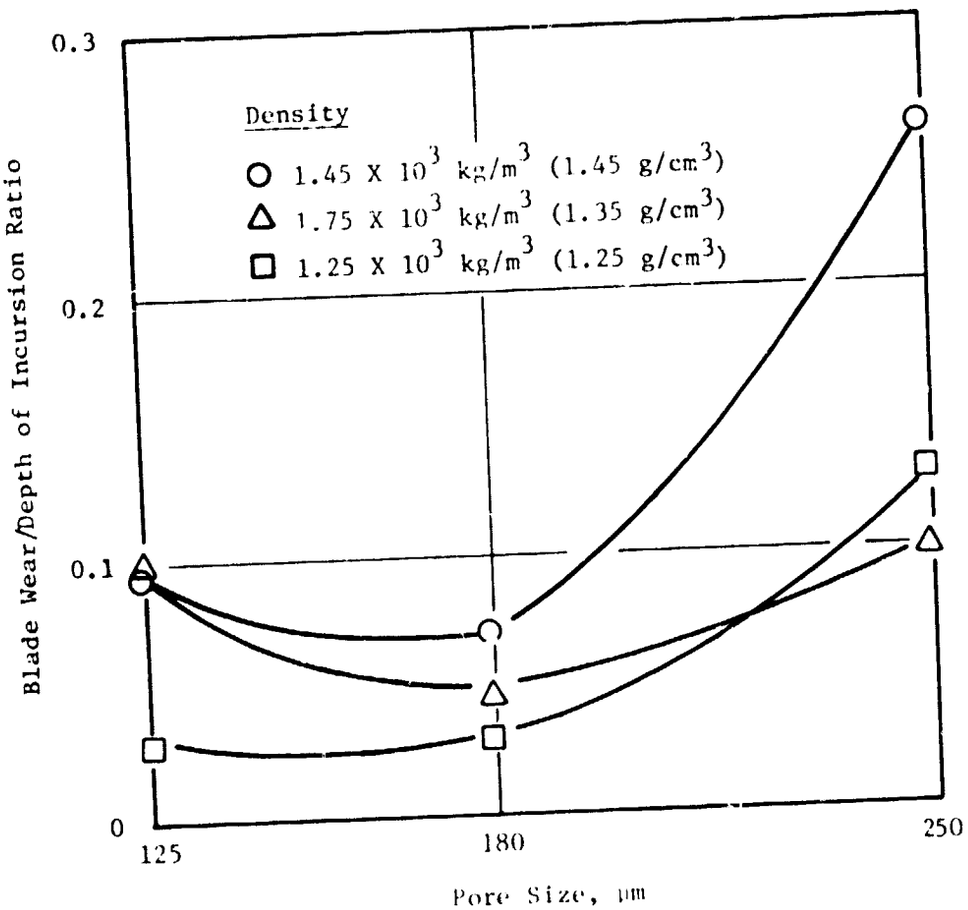


Figure 36. Effect of Pore Size on Blade Wear/Depth of Incursion Ratio in BN-Free Material.

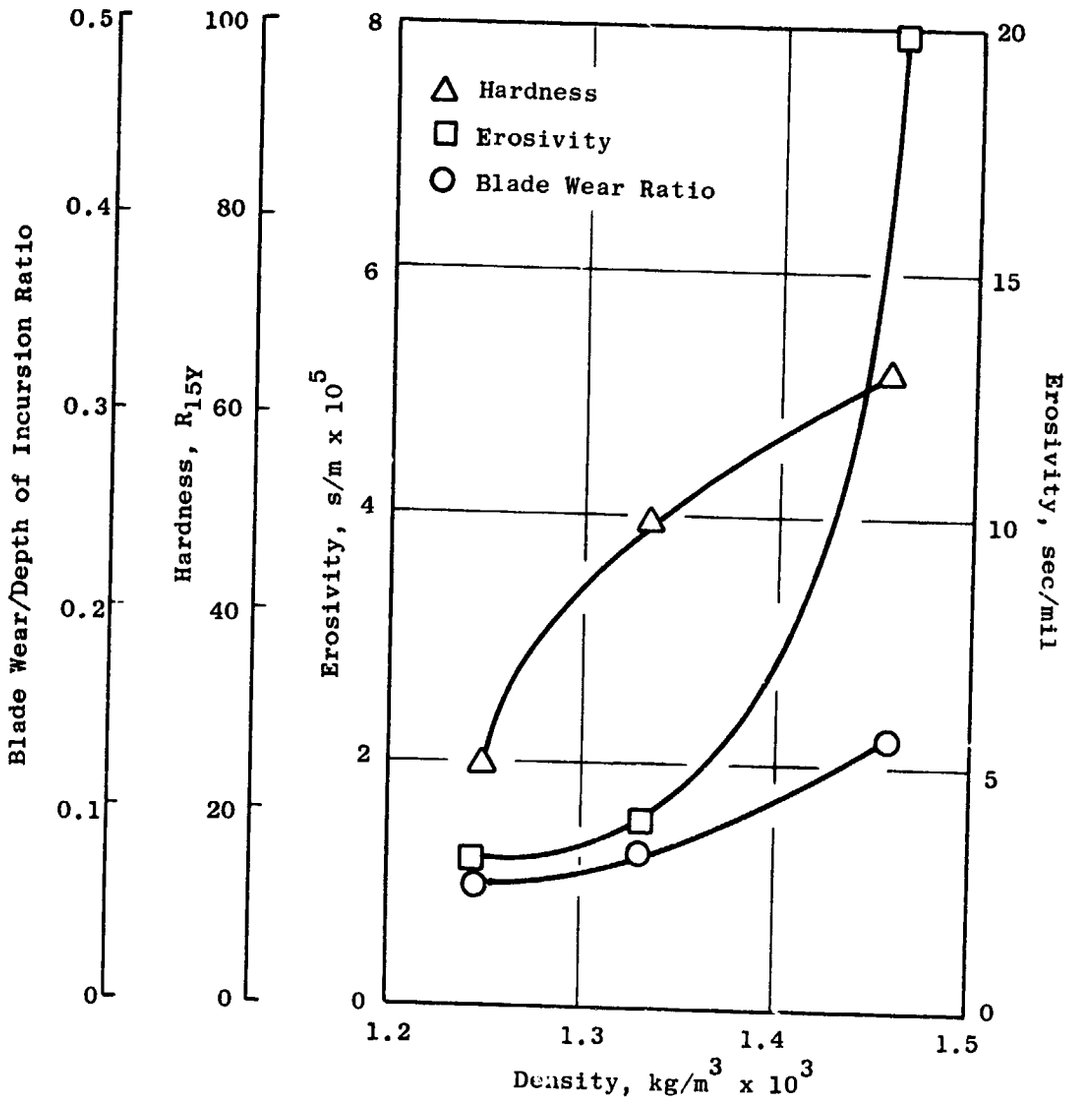


Figure 37. Overall Effect of Density on Surface Hardness, Erosivity, and Blade Wear Ratio in BN-Free Materials.

were averages of four erosion tests on each specimen. The test data indicated that the surface hardness, erosion resistance, and blade wear/depth of incursion ratio increased with an increase in the density of the BN-free material. The increase in pore size increased erosion resistance and blade wear/depth of incursion ratio and decreased surface hardness. Also, the effect of density variation on the properties was more significant than the effect of pore size variation.

1644K (2500° F) Static Oxidation Test Results

The 500-hour 1644 K (2500° F) static oxidation exposure test was completed. Weight, dimensional, and visual changes were recorded at 100-hour intervals. Plots of weight gain versus exposure time are shown in Figures 38 through 40. The oxidation weight gain for a BN-containing layer (without the substrate block) with SOA + 3 g BN additive level is shown in Figure 40 for comparison. Figure 41 shows a typical microstructure of a BN-free material of 1.45 x 10^3 kg/m^3 (1.45 g/cm^3) density after 500 hours of exposure at 1644 K (2500° F).

The following observations were made from the oxidation test:

- No melting or disintegration of specimens occurred.
- Specimen weight and dimensions increased in all the specimens.
- The percentage weight gain was higher in specimens of lower density and smaller pore sizes (for the same density).

The oxidized specimens were further evaluated by surface hardness measurements, cold particle erosion tests, and ballistic impact tests. The oxidation exposure increased the surface hardness of all the specimens and decreased the cold particle erosion resistance for most of the specimens (Table 13). The metallographic examination revealed the disappearance of some of the thin Si/SiC connecting links in the Si/SiC network via oxidation which explains the decrease in cold particle erosion resistance after oxidation exposure.

Ballistic Impact Test Results

Ballistic impact tests were made on all the variations in the statically oxidized condition (1644 K [2500° F], 500-hour exposure). The depth and diameter of the impact crater and cracks in the layer, if any, were recorded. The observations (Table 13) indicated that impact resistance generally increased with increasing density and decreasing pore size (for the same density). Figure 42 shows ballistic impact craters on two specimens which were ranked the best and the worst.

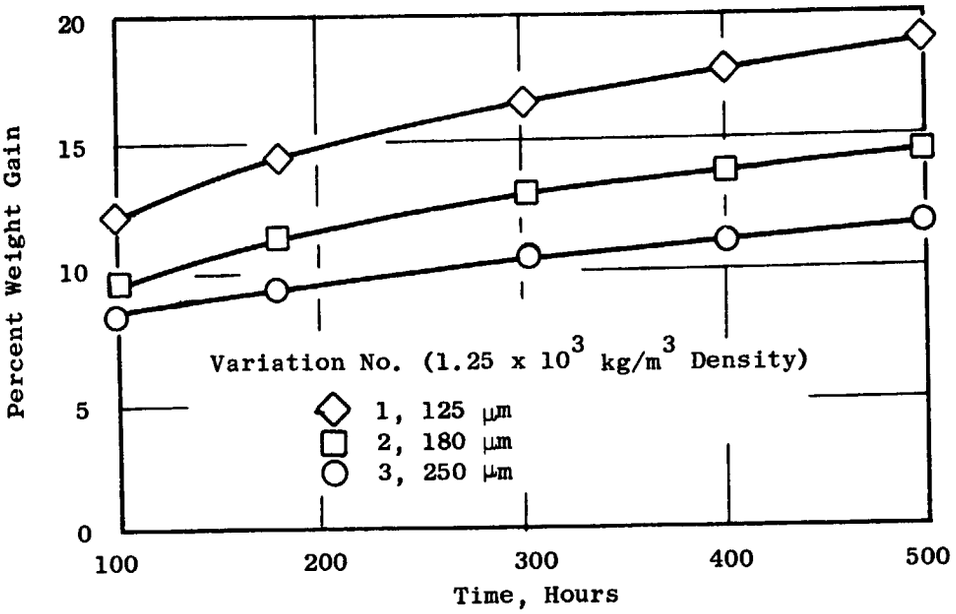


Figure 38. Percent Oxidation Weight Gain After Static Oxidation of BN-Free Material Variations 1 Through 3.

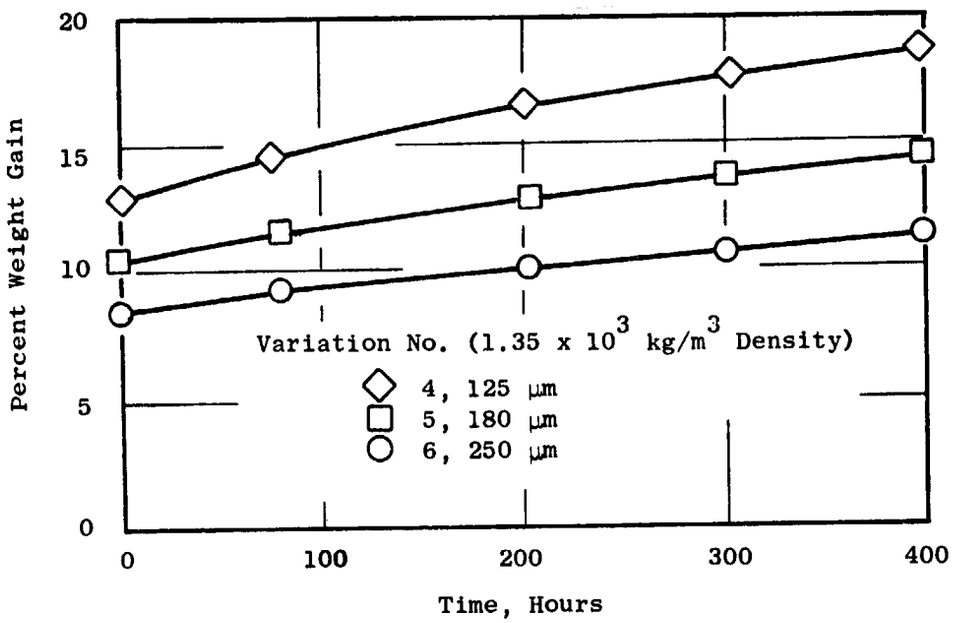


Figure 39. Percent Oxidation Weight Gain After Static Oxidation of BN-Free Material Variations 4 Through 6.

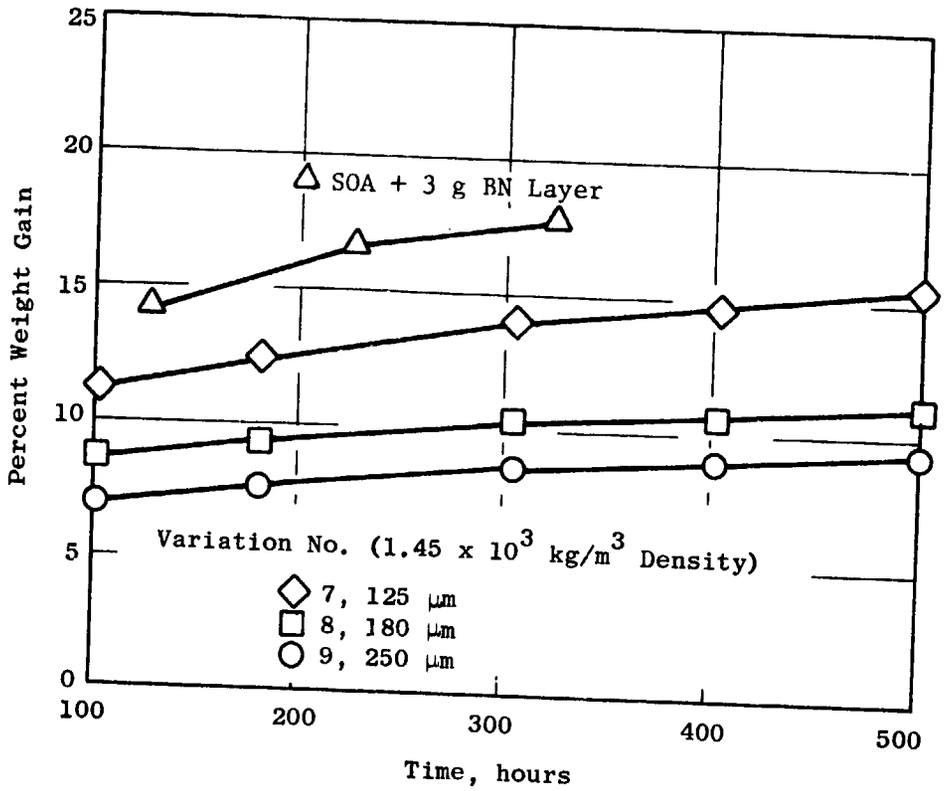


Figure 40. Percent Oxidation Weight Gain After Static Oxidation of BN-Free Material Variations 7 Through 9.

- The black areas are pores, the light gray areas are SiC, the white areas are Si, and the dark gray areas inside the SiC are unreacted carbon. The SiO_2 scale surrounds the Si/SiC network.

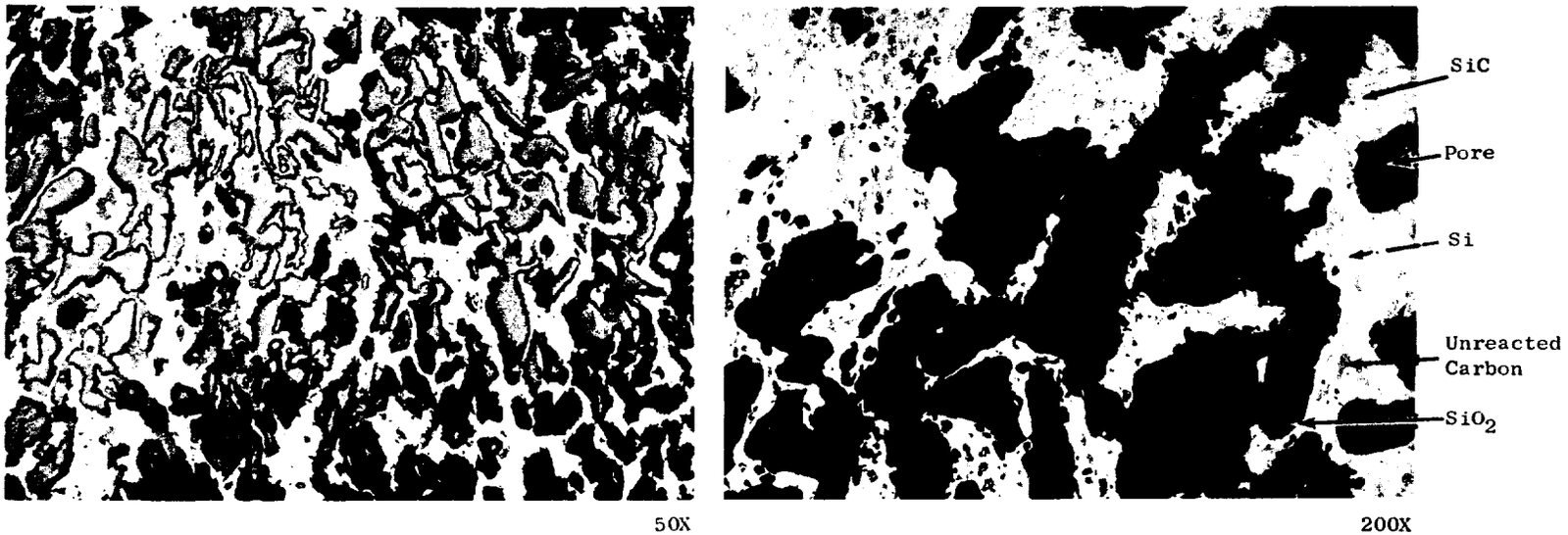
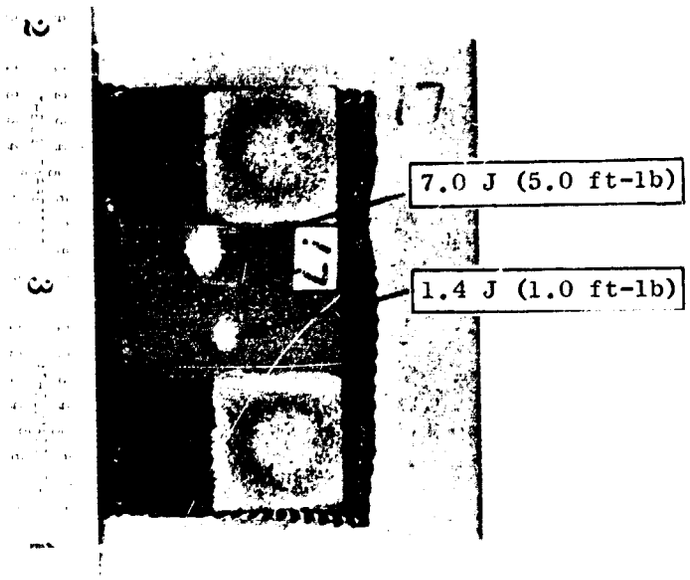


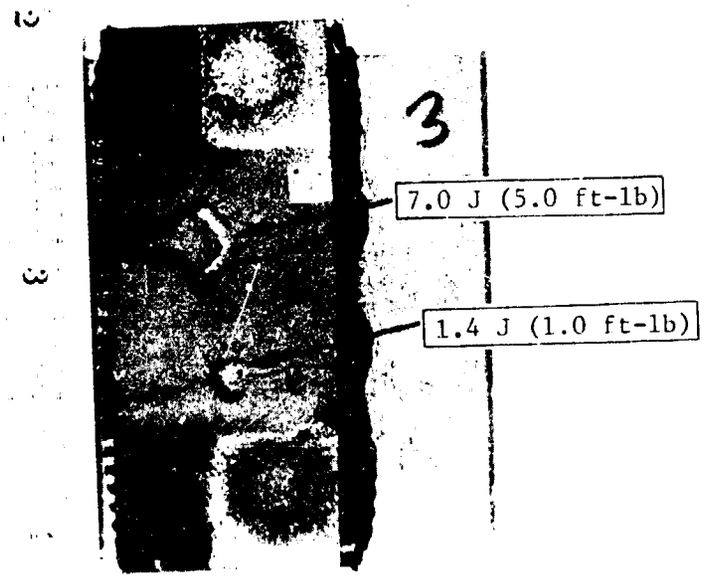
Figure 41. Typical Microstructure of a Cross Section of a BN-Free Material of 1.45 x 10^3 kg/m^3 (1.45 g/cm^3) Density and 250 μm Pore Size After Static Oxidation Exposure at 1644 K (2500° F) for 500 Hours.

Table 13. Preliminary Screening Tests Data on BN-Free Materials After 1644 K (2500° F) 500-Hour Oxidation.

Variation No.	Hardness R_{15Y}	Erosion Depth		Erosivity Factor		1 ft-lb Ballistic Impact Crater				5 ft-lb Ballistic Impact Crater			
						Diameter		Depth		Diameter		Depth	
		$m \times 10^{-4}$	(mil)	$s/m \times 10^{4}$	(mil)	$m \times 10^{-3}$	(mil)	$m \times 10^{-3}$	(mil)	$m \times 10^{-3}$	(mil)	$m \times 10^{-3}$	(mil)
1	74	5.94	(23.4)	7.5	(1.9)	3.81	(150)	1.4	(56)	5.33	(210)	3.63	(143)
1	68	5.64	(22.2)	7.9	(2.0)	3.43	(135)	1.0	(40)	6.35	(250)	3.07	(121)
2	50	7.77	(30.6)	5.9	(1.5)	4.57	(180)	1.8	(72)	8.64	(340)	3.56	(140)
2	56	4.55	(17.9)	9.9	(2.5)	4.83	(190)	1.2	(46)	6.35	(250)	3.35	(132)
3	58	1.73	(6.8)	26.0	(6.7)	4.06	(169)	1.3	(50)	6.48	(255)	2.90	(114)
3	71	2.97	(11.7)	4.2	(3.9)	3.94	(155)	1.3	(50)	6.73	(265)	2.84	(112)
4	89	5.70	(23.5)	7.5	(1.9)	3.81	(150)	0.8	(33)	7.24	(285)	3.35	(132)
4	88	4.75	(18.7)	9.5	(2.4)	3.43	(135)	0.8	(32)	5.72	(225)	3.18	(125)
5	75	4.14	(16.3)	11.0	(2.8)	4.06	(160)	1.1	(44)	6.10	(240)	3.15	(124)
5	78	4.24	(16.7)	11.0	(2.7)	4.06	(160)	0.9	(37)	6.10	(240)	2.67	(105)
6	65	2.16	(8.5)	21.0	(5.3)	3.81	(150)	1.3	(50)	6.35	(250)	3.38	(133)
6	74	2.06	(8.1)	22.0	(5.6)	3.81	(150)	1.1	(42)	5.46	(215)	2.92	(115)
7	90	4.75	(18.7)	9.5	(2.4)	2.67	(105)	0.7	(26)	5.33	(210)	1.37	(54)
7	90	5.00	(19.7)	9.1	(2.3)	2.67	(105)	0.7	(27)	5.33	(210)	1.85	(73)
8	85	2.13	(8.4)	21.0	(5.4)	3.18	(125)	0.7	(29)	1.04	(200)	2.59	(102)
8	86	2.59	(10.2)	17.0	(4.4)	2.79	(110)	0.7	(26)	5.59	(220)	1.65	(65)
9	83	2.64	(10.4)	17.0	(4.3)	3.56	(140)	0.7	(26)	5.08	(200)	2.79	(100)
9	79	2.44	(9.6)	19.0	(4.7)	3.30	(130)	0.8	(31)	5.59	(220)	2.41	(95)



Variation 7 Showing the Best
Impact Resistance (Rank 1)



Variation 2 Showing the Worst
Impact Resistance (Rank 9)

Figure 42. Ballistic Impact Resistance Test Results on BN-Free Materials.

Summary of Initial Screening Test Results on BN-Free Material Systems

1. None of the BN-free material variations showed any blade material transfer (scabbing) in the abradability test.

2. The surface hardness, cold particle erosion resistance, oxidation resistance, blade wear/depth of incursion ratio, and ballistic impact resistance increased with an increase in the density of BN-free materials.

3. The increase in pore size (for the same density) increased erosion resistance, oxidation resistance, and blade wear/depth of incursion ratio and decreased surface hardness and ballistic impact resistance.

4. The effect of density variation was more significant than the effect of pore size variation.

5. Overall, the BN-free materials showed better cold particle erosion resistance, abradability and oxidation resistance compared to BN-containing surface layers from Task I. Also, BN-free materials showed less scatter in property data compared to the BN containing surface layers.

Selection of the Three Most Promising BN-Free Material Candidates

The selection was based on the comparison of screening test data of each variation against "Musts" and "Wants" requirements. These requirements were established to meet the overall objectives of a turbine tip seal material for advanced aircraft engines. The "Musts" requirements are:

- No blade material transfer (scabbing) in abradability test.
- No melting or disintegration of specimen in oxidation test.

The "Wants" requirements are:

		Weighing Factor
1.	Good erosion resistance in the as-fabricated condition	10
2.	Good erosion resistance in the oxidation condition	8
3.	Good oxidation resistance	8
4.	Good ballistic impact resistance	8
5.	Low blade wear in the abradability test	5
6.	Good surface finish after rub	5

Each "Wants" requirement was given a weighing factor based on its importance. The test score of each variation was multiplied by the weighing factor to give the weighted score for each "Wants" requirement. The variation ranked the best in each "Wants" requirement was given a test score of 10; the test scores of other variations were then calculated from the test data. The weighted score of each variation is tabulated in Table 14 which also shows the total score for each variation. This analysis was not used in the selection of the most promising candidate in Task I because of a large scatter in property data in BN-containing surface layers.

Selection Results

All the variations met the "Musts" requirements. Based on the "Wants" requirements, Variations 9, 8, and 7 were ranked 1, 2, and 3, respectively (Table 14). In a second analysis in which each "Wants" requirement was given an equal weighing factor of 10, Variations 8, 9, and 7 were ranked 1, 2, and 3, respectively. These variations have the highest density 1.45 x 10^3 kg/m^3 (1.45 $\pm$ 0.05 g/cm^3) of all the variations evaluated and pore sizes of 125, 180, and 250 µm.

4.4 TASK II - ADVANCED SCREENING

Hot Rub Tests

Rub tests were conducted on the three most promising specimens (7, 8, and 9, Table 2) at 894 K (1150° F) to 950 K (1250° F). The results of the rub tests are shown in Table 15; Figure 43 shows a representative specimen after the rub.

The important rub test observations are as follows:

1. All the specimens rubbed well with no objectional blade metal transfer (scabbing). There was no significant difference between the rubbed surfaces of specimens with varying pore sizes.

2. Specimens with higher surface hardness (R_{15Y}) showed greater blade wear to depth of rub ratio.

3. The temperature at the back of some of the specimens (3.1 x 10^{-3} m thick) rose to 1089 K (1500° F) due to frictional heat generated during the rub.

4. In all cases, the shear force during the rub was equal to or less than 3.0 N (0.65 lb).

Table 14. Comparison of Candidates Based on "Wants" Requirements.

"Wants" Requirements	Weighing Factor	Variation 1		Variation 2		Variation 3		Variation 4		Variation 5		Variation 6		Variation 7		Variation 8		Variation 9	
		Sc	Wt Sc	Sc	Wt Sc	Sc	Wt Sc	Sc	Wt Sc	Sc	Wt Sc	Sc	Wt Sc	Sc	Wt Sc	Sc	Wt Sc	Sc	Wt Sc
Good Erosion Resistance in the as-fabricated condition	10	0.7	7	1.1	11	1.3	13	1.2	12	1.2	12	1.6	16	5	50	6	60	10	100
Good erosion resistance in the oxidized condition	8	3.7	30	3.7	30	9.8	78	5	40	4	32	10	80	4.3	35	9	72	8.3	66
Good oxidation resistance	8	5.1	40	6.5	52	8.1	65	5.1	41	6.6	53	8.4	68	6.1	49	8.4	67	10	80
Good ballistic impact resistance at 5 ft-lb	8	5.5	44	5.3	42	4.5	36	8.1	65	6.5	52	5.7	46	10	80	9.6	77	9.3	74
Good ballistic impact resistance at 5 ft-lb	8	7.6	60	6.3	50	7.3	59	7.1	57	7.7	61	7.7	61	10	80	9.3	75	8.7	70
Low blade wear in the abradability test	5	10	50	10	50	2.3	12	3	15	6.6	33	3	15	3.3	16	4.2	21	1.1	6
Good surface finish after rub	5	8	40	5	25	3	15	10	50	6	30	2	10	10	50	7	35	4	20
Total Score		271		260		278		280		273		296		360		407		416	
Rank		8		9		6		5		7		4		3		2		1	
Total Score With weighing factor of 10 for each desired requirement		406		379		363		395		386		384		487		535		514	
Rank		4		8		9		5		6		7		3		1		2	

Legend

Sc = Test Score
Wt Sc = Test Score x Weighing Factor
Best Rank = 1

Table 15. Results of Hot Rub Tests on BN-Free Materials.

Specimen No.	Density		Pore Size,	Surface Hardness	Rub Temperature				Rub Force		Depth of Rub		Blade Wear		Ratio of Blade Wear to Depth of Rub	Scab
					Start		Maximum									
	$kg/m^3 \times 10^3$	(g/cm^3)	μm	R_{15Y}	K	(° F)	K	(° F)	N	(lb)	$m \times 10^{-5}$	(mil)	$m \times 10^{-5}$	(mil)		
1	1.43	(1.43)	250	66	922	(1200)	1033	(1400)	2	(0.5)	1.0	(40)	5	(2)	0.05	No, some layer smearing
2	1.44	(1.44)	250	69	927	(1210)	933	(1220)	<1	(<0.3)	0.8	(31)	12	(5)	0.16	No, some layer smearing
3	1.44	(1.44)	180	78	950	(1250)	1089	(1500)	2.9	(0.65)	1.1	(44)	20	(8)	0.18	Some spotty blade transfer, some layer smearing
4	1.43	(1.43)	180	75	936	(1225)	991	(1325)	<2	(<0.5)	0.6	(24)	12	(5)	0.21	Some spotty blade transfer, some layer smearing
5	1.46	(1.42)	120	63	936	(1225)	1005	(1350)	2	(0.5)	1.3	(50)	10	(4)	0.08	No, some layer smearing
6	1.47	(1.44)	120	67	894	(1150)	1060	(1450)	2	(0.5)	0.3	(10)	3	(1)	0.10	No, some layer smearing

Specimen 6, Table 15

Figure 43. Advanced Screening BN-Free Rub Specimen After the Hot Abradability Test.

Bonding Study

Mach 1.0 gas oxidation/erosion test on BN-free rub layer material systems could not be carried out because of the lack of a technique to attach the layers to a solid ceramic block necessary to conduct the test. Therefore, the Task II Technical Plan was revised to develop a brazing technique for attaching the layers to a dense ceramic substrate block by using high-purity silicon. The technique suggested by NASA-Lewis/Solar and developed under contract NAS3-20081 consisted of the following steps:

1. Roughen the HP SiC or the HP Si_3N_4 surface on a 165-grit diamond paper, clean with solvent, and dry.

2. Clean and dry the BN-free layers.

3. Apply Nicrobraze 500 to both contact surfaces.

4. Sprinkle silicon uniformly before the Nicrobraze dries; the number of Nicrobraze and silicon applications determines the thickness of Si powder.

5. Record weights and thicknesses.

6. Place in the furnace with a small amount of pressure to assure contact.

7. Heat to 1755 K (2700° F), keep at temperature for 5 minutes under vacuum.

Table 16 summarizes all the experiments to braze the BN-free layer to a hot pressed silicon carbide or a hot pressed silicon nitride block. The metallographic examination (Figure 44) of a BN-free layer bonded to a hot-pressed SiC block showed a metallurgical bond in which the braze interface consisted of Si + SiC instead of pure Si. Specimens from the promising experiments 8992-1B and 8336-1B, survived thermal cycling from 1477 K (2200° F) and 1589 K (2400° F) but failed at low strengths 1.8 MPa (265 psi) and 3.3 MPa (480 psi), respectively, in tensile bond tests. Examination of the fractured surface after tensile testing, showed that the BN-free layer was bonded in only about one-fourth of the contact area near the perimeter of the HP SiC block (Figure 45). It was postulated that the partial bonding could be due to a thermal gradient within the HP SiC block because of a too rapid heating rate. In experiments 9038-1A, -1B, and -1D a slower heating rate was used to reduce any thermal gradient. The fracture surfaces of experiments 9038-1A and -1D showed wetting of the silicon across the entire surface rather than wetting at the perimeter only. However, the bond was not adequate as evidenced by early failures in thermal shock and bond tests.

In experiments 9064-1A, -1B, and -1C, a heating rate slower than that of experiments 9038-1A, -1B, and -1D was used. Specimen 9064-1A was heated to 1723 K (2642° F) while Specimen 9064-1B was heated to 1671 K (2552° F) and

Table 16. Results of BN-Free Layer Bonding Study.

Experiment No.	Block Material	Thickness of Silicon Powder				Results
		Block		Layer		
		m × 10^{-4}	(mil)	m × 10^{-4}	(mil)	
8263-1B	* HP SN	2.5	(10)	0.8	(3)	No bond.
8270-1A	HP SiC	2.8	(11)	1.0	(4)	Some bond, separated on prying.
8270-1B	HP SiC	6.6	(26)	2.0	(7)	Bond, 1/3 came off initially, remaining bond survived 10 cycles from 1477 K (2200° F) and 10 cycles from 1589 K (2400° F), 1/3 came off after the 6th cycle from 1589 K (2400° F).
8292-1B	HP SiC	9.9	(39)	2.0	(7)	Bond survived 10 cycles from 1477 K (2200° F) and 10 cycles from 1589 K (2400° F), no separation. Tensile strength = 1.82 MPa (265 psi) at room temperature (failure partially in the layer and partially in the braze interface).
8292-1C	HP SiC	6.1	(24)	2.0	(7)	Repeat of 8270-1B, some bond, separated on prying.
8324-1A	HP SiC	7.4	(29)	2.0	(8)	Repeat of 8270-1B, separated on prying.
8336-1A	HP SiC	9.7	(38)	2.0	(8)	Repeat of 8292-1B, bond separated during bond test setup.
8336-1B	HP SiC	10.0	(40)	2.0	(6)	Repeat of 8292-1B bond survived 10 cycles from 1477 K (2200° F) and 10 cycles from 1589 K (2400° F) no separation. Tensile strength = 3.31 MPa (480 psi) at room temperature (failure partially in the layer and partially in the braze interface).
8336-1C	HP SiC	10.0	(40)	2.0	(7)	Bond, separated on prying.
8336-1D	HP Sic	10.0	(40)	2.0	(6)	Bond, layer came off after 2 cycles from 1477 K (2200° F).
8336-1E	HP SiC	11.0	(43)	2.0	(6)	Bond separated during bond test setup.

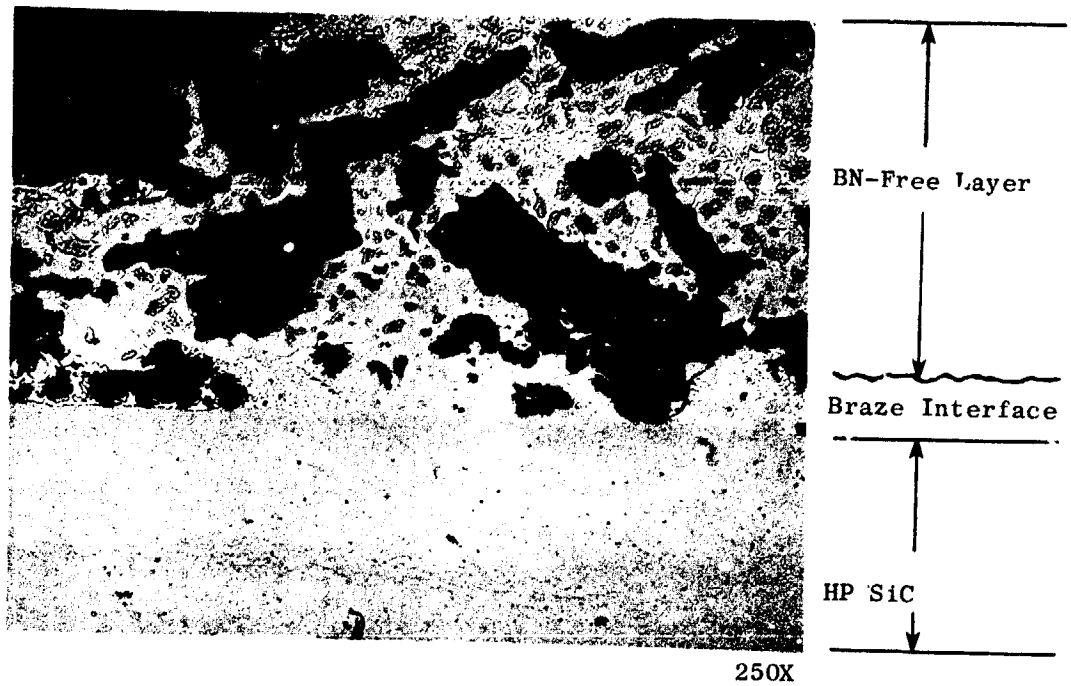
Experimental Conditions

Silicon particle size = -140 + 325 mesh
Silicon purity = 99.999%
Furnace temperature = 1755 K (2700° F)
Vacuum = 10^{-1} - 10^{-3} MPa (10^{-3} - 10^{-5} Torr)

*HP = Hot Pressed

Table 16. Results of BN-Free Layer Bonding Study (Concluded).

Experiment No.	Block Material	Thickness of Silicon Powder				Results
		Block		Layer		
		m × 10^{-4}	(mil)	m × 10^{-4}	(mil)	
9038-1A	HP SiC	10	(39)	2	(6)	Slow heating rate, bond separated during bond test setup.
9038-1B	HP SiC	10	(41)	2	(6)	No pressure applied during bonding, slow heating rate, did not bond.
9038-1C	HP SiC	10	(41)	2	(7)	No pressure applied during bonding, normal heating rate, did not bond. Silicon did not melt.
9038-1D	HP SiC	13	(50)	2	(9)	Slow heating rate. Bond survived thermal shock from 1477 K (2200° F), but failed during the first cycle from 1589 K (2400° F).
9064-1A	HP SiC	10	(39)	1	(5)	Slower heating rate to 1723 K (2642° F). Bond survived 10 cycles of thermal shock from 1477 K (2200° F), but failed during first cycle from 1589 K (2400° F).
9064-1B	HP SiC	10	(39)	2	(6)	Slower heating rate to 1673 K (2552° F). Bond failed during first cycle from 1477 K (2200° F).
9064-1C	HP SiC	10	(40)	2	(6)	Slower heating rate to 1693 K (2588° F). Layer stuck to the die, not the HP SiC block.



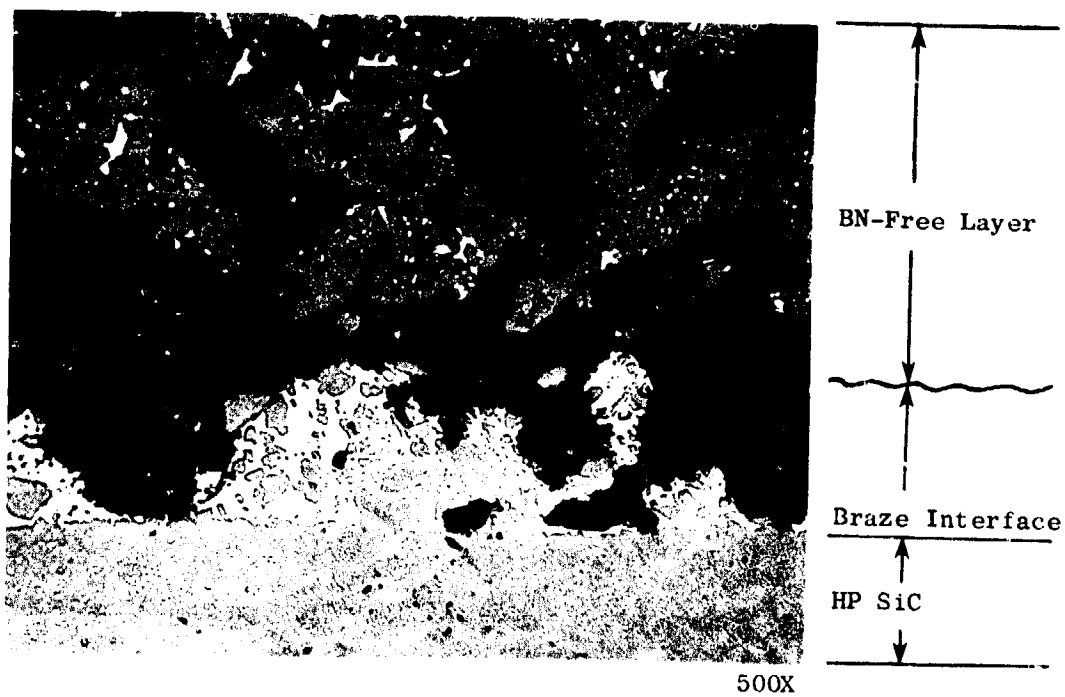


Figure 44. Typical Microstructure of Braze Interface Between the BN-Free Layer and an HP SiC Block.

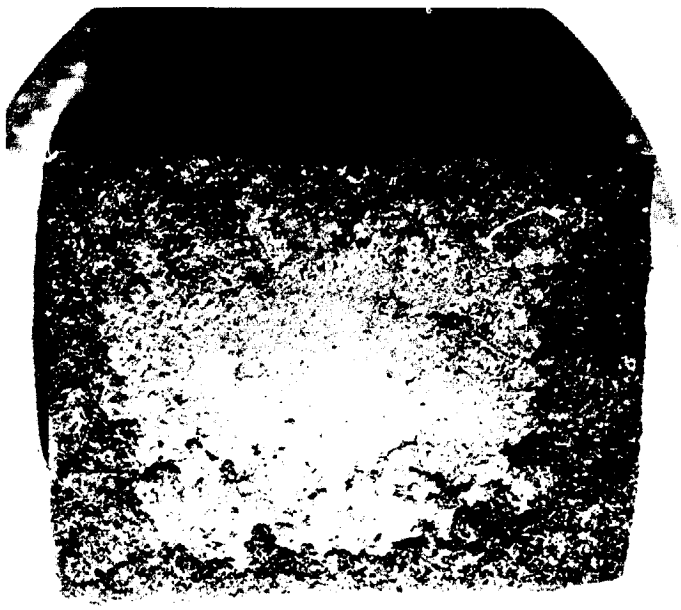


Figure 45. Specimen from BN-Free Bonding Experiment No. 8336-1B Showing Nonuniform Wetting of Si on the Hot Pressed SiC Block.

Specimen 9064-1C was heated to 1693 K (2588° F). The bonding in Specimen 9064-1A was improved from experiment 9038 specimens. Specimens 9064-1B and -1C showed poor bonding.

In summary, the experiments conducted in this program did not yield a reproducible bond. Further experimentation is necessary to improve the bond strength and the reproducibility of the technique.

4.5 TASK III - ADVANCED LABORATORY EVALUATION

Task III included specimen fabrication, Mach 1.0 gas oxidation/erosion test holder modification and checkout, and the 1644 K (2500° F) Mach 1.0 gas oxidation/erosion test of BN-containing layer specimens on hot pressed silicon carbide and Silcomp^{TM} substrates. During farication, cracks in the braze interface (between the surface layer and the hot pressed SiC substrate block), which sometimes propagated into the HP SiC block, occurred upon diamond and ultrasonic machining of the composite specimen. A modification to the specimen geometry and a slower furnace cooling-down cycle alleviated the specimen cracking problem. In addition, only ultrasonic machining was utilized.

Prior to the 1644 K (2500° F) Mach 1.0 gas oxidation/erosion test of ceramic specimens, it was necessary to modify and check out the holder. Modifications were necessary because:

- The holder showed signs of thermal distress and weld cracking.
- Specimens were cracked due to the holding mechanism. Subsequent analysis identified two possible causes for fracture of the specimens; (a) the colder metal holder did not accommodate expansion of the hotter ceramic specimen, and (b) uneven loading of the ceramic specimen at the base.

The following changes were incorporated in the test holder:

1. Add ZrO_2-coated HS188 metal spacers around all the contact sides of the ceramic specimen. The use of a plasma-sprayed zirconia coating was expected to (a) limit heat flow to the metal holder, (b) prevent chemical reactions between SiC and metal holder, and (c) provide compliance against point loading of the ceramic specimen.
2. Add ZrO_2-coated heat shield to minimize radiation losses.
3. Add IN718 springs on the loading side of the ceramic specimen to provide additional compliance against point loading of the ceramic specimens.

Six hot pressed silicon carbide blocks without the surface layers were used in the holder checkout test and carried out at the following conditions:

- Gas Velocity = Mach 1.0
- Holder Rotation = 400 rpm
- Cycle = 55 minutes hot and 5 minutes cold
- Temperature, K (° F) = 1366, 1422, 1477, 1533, 1589, 1644 K (2000, 2100, 2200, 2300, 2400, 2500° F)
- No. of Cycles = 5 at each temperature

The holder and all the specimens performed satisfactorily in the test except for some weld microcracks in the holder. Consequently, the holder was slightly modified for the subsequent tests. The IN718 springs which were placed on the loading side of three (out of six) ceramic specimens completely relaxed. Since they did not offer any advantage, they were not used in future tests.

The Mach 1.0 gas oxidation/erosion test was conducted at 1644 K (2500° F) with the layer containing specimens. Three of the six specimens had hot pressed silicon carbide; the other three had Silcomp™ as the substrate materials. As shown in Table 17 and Figure 46 five out of six BN-containing layers showed excessive layer erosion in only 2 to 5 hours of testing at 1644 K (2500° F). Also, on inspection after the third and the fifth cycles, two of the specimens were found to have fractured, and the layers from two specimens spalled off at Cycle 3. It was noted that the two specimens had fractured in the area exposed to the high-velocity gas stream, and not in the areas where the specimens were held in the metal holder, indicating the adequacy of the test holder. The test was stopped after 5 hours of testing. After the oxidation test, the specimens were evaluated for surface hardness (R_{15Y}), room temperature abradability, and cold particle erosion resistance.

A second test with five new specimens (one continued from 1644 K [2500° F] testing) was conducted at 1589 K (2400° F). All the specimens showed unacceptable layer erosion at this temperature after eight 1-hour cycles. Consequently, the test temperature was lowered to 1533 K (2300° F). The results of the test at 1584 K (2400° F) and 1533 K (2300° F) are summarized in Table 18. Figures 47 through 50 show conditions of the specimens at various stages of the 1589 K/1533 K (2400°/2300° F) test. After oxidation exposure, the specimens were evaluated for surface hardness, room temperature abradability, and cold particle erosion resistance.

The following conclusions were drawn from the first and second Task III Mach 1.0 gas oxidation tests:

- The BN-containing layers showed inadequate and variable gas erosion resistance at temperatures as low as 1533 K (2300° F).
- The Silcomp™ interface which was used to braze the surface layer to the HP SiC block showed an erosion loss due to gas erosion/oxidation at 1533 K (2300° F).

Table 17. Test Results After Five 1-Hour Cycles at 1644 K (2500° F) in First Task III Mach 1.0 Gas Oxidation/Erosion Test.

Identification	Layer Composition	Substrate Block	Layer Surface Hardness (R_{15Y})			Test Results
			Avg of 9 Readings	High Value	Low Value	
35F1B	SOA 1976	**HP SiC	77	83	68	100% of layer eroded over one-half of the specimen length,* and layer spalled off at Si/SiC interface at Cycle 3.
23GA2	SOA + 3 g BN	HP SiC	70	78	63	Specimen fractured after Cycle 5 layer spalled off at Si/SiC interface at Cycle 3.
23GB1	SOA + 3 g BN	HP SiC	69	80	42	50% of layer eroded over 20% of the specimen length.
7F3A	SOA 1976	Silcomp	61	80	40	No erosion.
16FA1	SOA 1976	Silcomp	49	66	32	50% of the layer eroded over 12% of the specimen length.
15F2A	SOA 1976	Silcomp	46	74	11	50% of the layer eroded over 30% of the specimen length; specimen fractured at Cycle 3.

* Erosion started at the tip of the specimen and gradually extended toward the base with exposure time.

** Hot pressed.

Figure 46. First Task III Mach 1.0 Gas Oxidation/Erosion Specimens After 5 Hours at 1644 K (2500° F).

Table 18. Test Results After Eight 1-Hour Cycles at 1589 K (2400° F) and Twenty 1-Hour Cycles at 1533 K (2300° F) in Second Task III Mach 1.0 Gas Oxidation/Erosion Test.

Sample Identification	Layer Compositon	Substrate Block	Layer Surface Hardness (R_{15Y})			Test Results
			Average of 9 Readings	High Value	Low Value	
35F1A	SOA 1976	HP SiC(a)	71	81	53	100% of layer eroded over one-half of the specimen length**, and layer spalled off at Si/SiC interface at Cycle 28.
15F28	SOA 1976	Silcomp	64	79	35	90% of layer eroded over 20% of the specimen length, Silcomp eroded at tip of the specimen.
23GB1*	SOA + 3 g BN	HP SiC	69	84	42	95% of the layer eroded over 25% of the specimen length.
16FA2	SOA 1976	Silcomp	43	57	31	80% of the layer eroded over 30% of the specimen length.
23GA1	SOA + 3 g BN	HP SiC	72	81	59	100% of the layer eroded over 37% of the specimen length.
24FB2	SOA + 3 g BN	Silcomp	40	72	22	95% of the layer eroded over 37% of the specimen length.

*Sample also ran 5 cycles at 1644 K (2500° F).

**Erosion started at the tip of the specimen and gradually extended towards the base with exposure time.

(a)Hot Pressed

Figure 47. Second Task III Mach 1.0 Gas Oxidation/Erosion Specimens in Holder After 8 Hours at 1589 K (2400° F) and 20 Hours at 1533 K (2300° F).

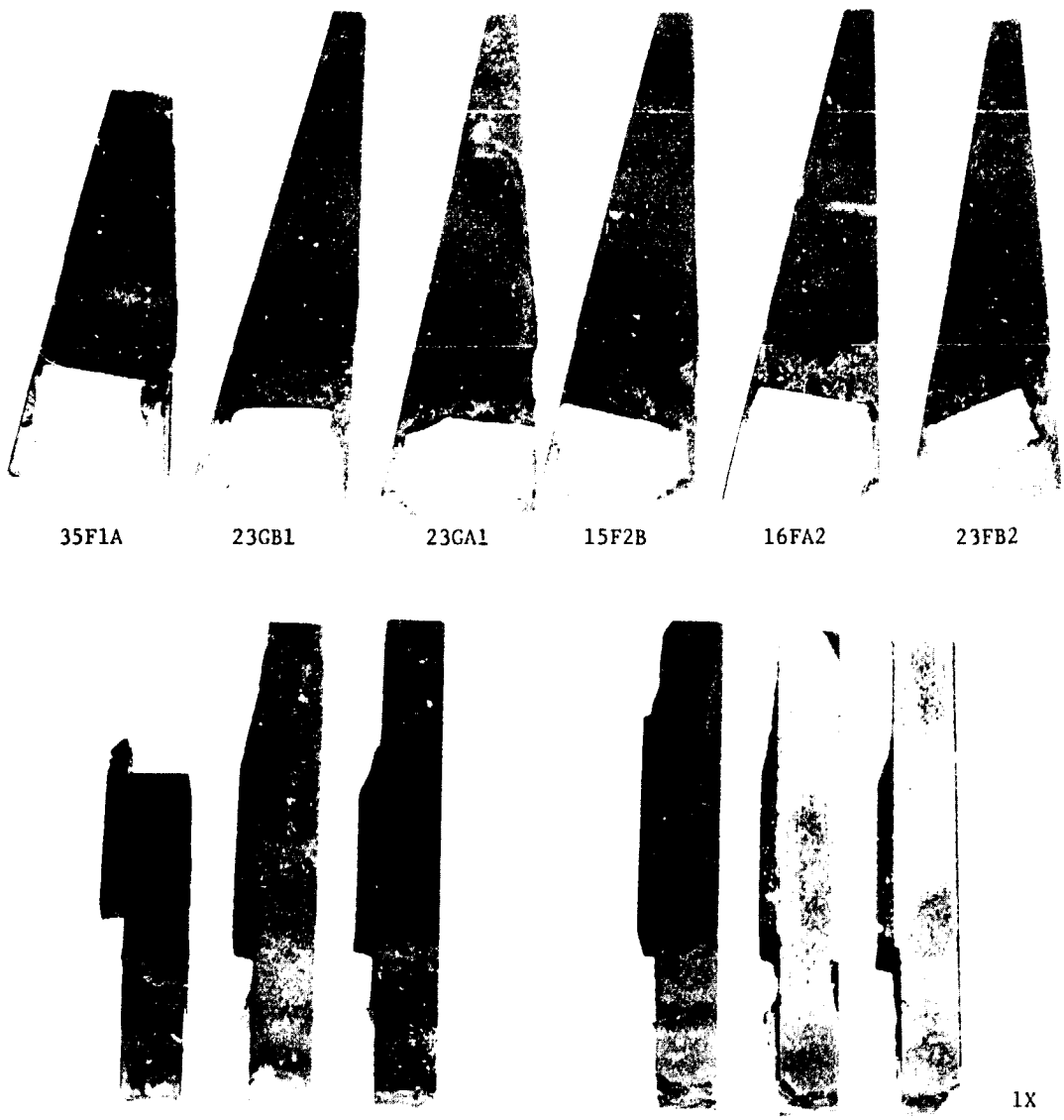


Figure 48. Second Task III Mach 1.0 Gas Oxidation/Erosion Specimens After 8 Hours at 1589 K (2400° F) and 20 Hours at 1533 K (2300° F).

5 Hours at 1589 K (2400° F)

8 Hours at 1589 K (2400° F)

8 Hours at 1589 K (2400° F)
12 Hours at 1533 K (2300° F)

8 Hours at 1589 K (2400° F)
20 Hours at 1533 K (2300° F)

Figure 49. Second Task III Mach 1.0 Gas Oxidation/Erosion Specimen 16FA2 at Various Stages of Testing at 1589/1533 K (2400°/2300° F).

Figure 50. Closeup View of Task III Mach 1.0 Gas Oxidation Specimen 23GA1 After 8 Hours at 1589 K (2400° F) and 10 Hours at 1533 K (2300° F).

- The bond of the BN-containing layer to the HP SiC block was marginal, as evidenced by layer spallation.

- No definite correlation exis. d between the surface layer hardness and hot gas erosion.

- Melting of free silicon occurred in the laver, the Silcomp^{TM} interface, and the Silcomp^{TM} substrate block.

The exposure of the BN-containing abradable layers to the hot gas environment affected hardness, rub characteristics, and the cold particle erosion resistance. Though there was a large scatter in these data, the surface hardness of the layers (R_{15Y}) increased by about 10 points. The increased hardness resulted in improved cold particle erosion resistance. In abradability tests, the specimens had a slight tendency for scabbing (blade material transfer).

The results of the first and the second Task III Mach 1.0 gas oxidation/erosion tests were different from the results of the Task I test which showed that the material was capable of surviving 84 hours at 1477 K (2200° F) and 16 hours at 1589 K (2400° F). Based on the tests conducted under this program and under various GE programs, the hot gas erosion resistance had previously been related to the average surface hardness (R_{15Y}) of the BN-containing rub layers. Layers with average surface hardness below 50 experienced hot gas erosion, while layers with hardnesses between 55 and 91 did not erode. However, in rub tests conducted under in-house GE programs, the harder layers with $R_{15Y} > 75$ caused blade material transfer (scabbing). Thus, a hardness range of R_{15Y} 60 $\pm$ 10 was selected. The Task III Mach 1.0 tests were intended to evaluate specimens made to span this hardness range and to generate longer-time gas erosion data at 1644 K (2500° F). The 11 specimens used in the test had surface hardnesses (R_{15Y}) ranging from 46 to 77.

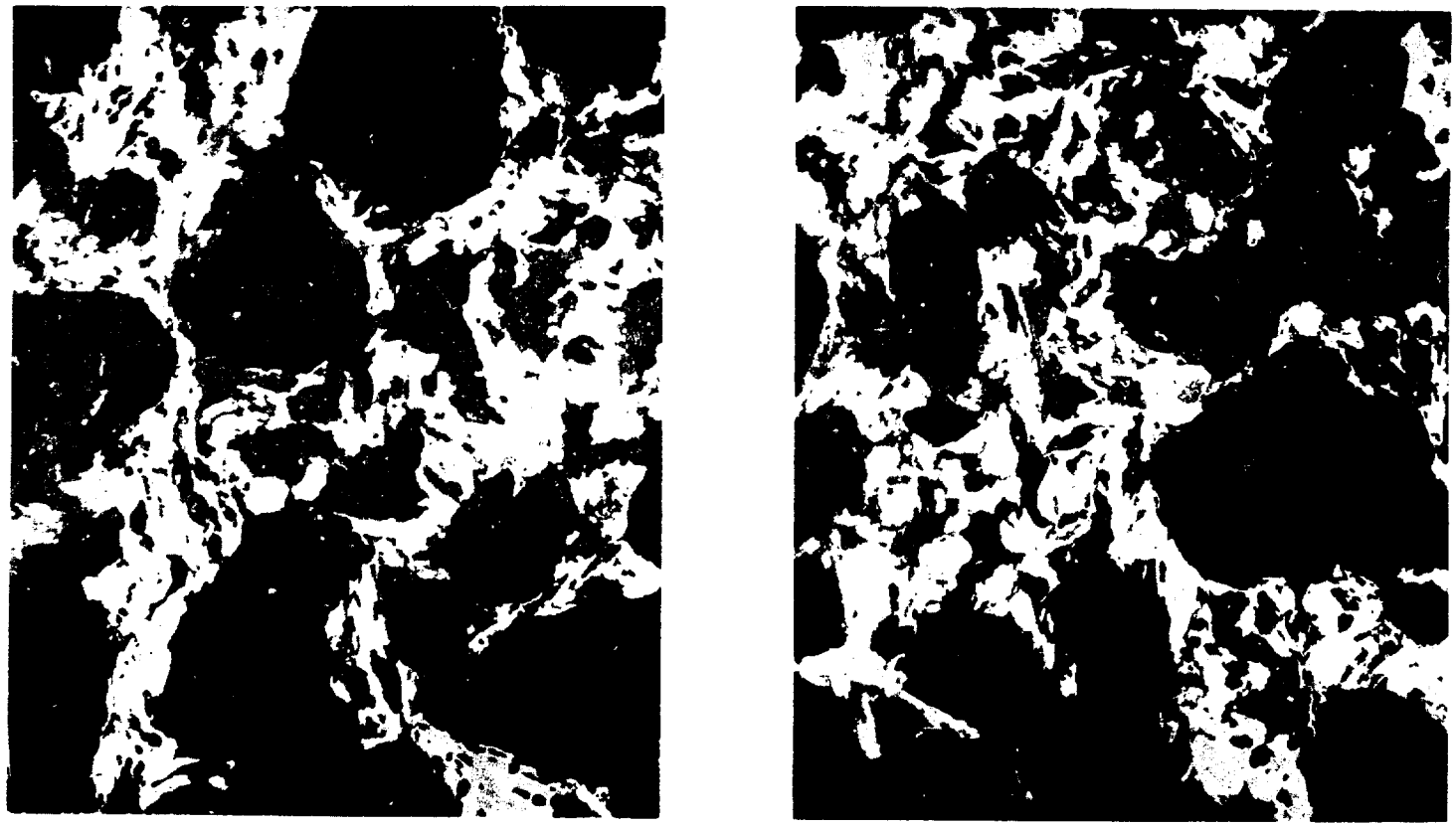
In attempting to explain why the results of the first and the second Task III Mach 1.0 gas oxidation/erosion tests conducted at 1533 K (2300° F) to 1644 K (2500° F) were different from the Task I test, it was postulated that preoxidation of the layers could improve the hot gas erosion resistance of the layers because the microstructure of the Task I Mach 1.0 gas oxidation/erosion test specimens showed a continuous oxide film around the Si/SiC network whereas the Task III Mach 1.0 test specimens showed either the absence of or a discontinuous oxide film (Figures 51 and 52).

A third Task III Mach 1.0 gas oxidation/erosion test was conducted using new specimens. Details of the test are given below.

- Gas Velocity — Mach 1.0
- Holder Rotation — 350 rpm
- Cycle — 55 minutes hot, 5 minutes cold
- Temperature, K (° F) — 1477 K, 1533 K, 1589 K (2200, 2300, 2400° F)

Figure 51. Typical Microstructure of a Specimen After Test I Mach 1.0 Gas Oxidation/Erosion Test at 1473 K (2200° F) Showing Continuous SiO_2 Film Around the Si/SiC Network.

C-2



200X 200X

Figure 52. Typical Microstructure of a Specimen After the First Task III Mach 1.0 Gas Oxidation/Erosion Test at 1644 K (2500° F) Showing the Absence of SiO_2 Film Around the Si/SiC Network.

- No. of Cycles 15 at each temperature

The temperature was monitored by a two-color pyrometer sighted on the rotating ceramic specimens. Ultrasonic scans (Figure 53) of the surface layers attached to the substrate blocks were made prior to the test to detect any unjoined area that could lead to layer spallation which occurred in the first and the second Mach 1.0 gas oxidation/erosion tests. In the scan, a dark area signifies poor bonding. The test was conducted initially at 1477 K (2200° F) for 15 hours to preoxidize the specimens, and then the temperature was raised to 1533 K (2300° F) and eventually to 1589 K (2400° F) for 15 hours each, for a total of 45 hours of operating time. Two statically oxidized specimens at 1366 K (2000° F) for 65 hours were also used to evaluate the effect of prior static furnace exposure. The plan to test the specimens at 1644 K (2500° F) could not be carried out because the refractory liner in the specimen chamber was excessively eroded. The results of the oxidation test and the post test evaluations are given in Table 19.

Photographs of several specimens are shown at various stages of Mach 1.0 gas oxidation/erosion testing in Figures 54 through 56. Observations made concerning the erosion behavior of the BN containing rub specimens in this test are summarized below:

1. The specimens showed a significant improvement in the hot gas erosion resistance compared to the first and the second Task III Mach 1.0 gas oxidation/erosion tests.

2. The specimens showed no erosion after running at 1477 K (2200° F).

3. Slight erosion initiated at 1533 K (2300° F); the erosion was evident in slight rounding of previously square edges and in increased surface roughness of the layer surface. Erosion continued at 1589 K (2400° F), but the rate was not different from the 1533 K (2300° F) test.

4. The erosion behavior of the specimens preoxidized statically at 1366 K (2000° F) was not significantly different from those which were not preoxidixed in situ.

5. The microstructural examination shows SiO_2 film around some of the Si/SiC network (Figure 57).

Posttest evaluation indicated that the surface hardness of the layers increased by 15 to 22 points due to oxidation exposure. Therefore, the surface hardness (R_{15Y}) of the layers ranged from 78 to 94. The room temperature rub tests showed large blade wear to depth-of-incursion ratios and blade material transfer in all the specimens indicating unacceptable rub properties of the exposed specimens (Figure 58). Cold particle erosion and room temperature rub test results on as-fabricated specimens are also shown in Figure 58 for comparison. This observation was consistent with observations made under in-house GE programs that rub layers with surface hardness (R_{15Y}) greater

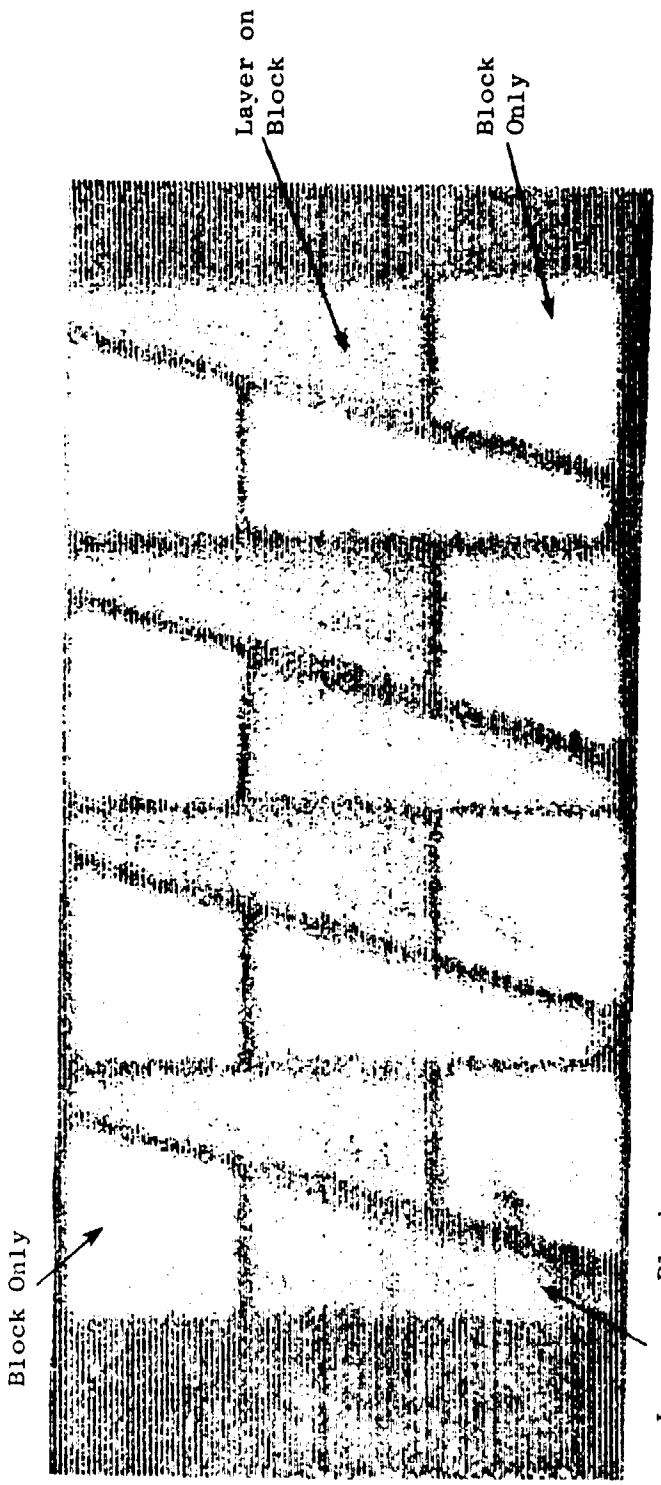


Figure 53. Ultrasonic Scan Results of Third Task III Mach 1.0 Gas Oxidation/Erosion Specimens.

Table 19. Hardness, Abradability, and Cold Particle Erosion Test Results After 1477/1533/1589 K (2200°/2300°/2400° F) Third Task III Mach 1.0 Gas Oxidation/Erosion Exposure.

Sample Identification	Layer Composition	Substrate Block	Layer Surface Hardness After Exposure, R_{15Y} (1)	Layer Surface Hardness After Exposure, R_{15Y} (1)	Blade Wear/ Depth of Incursion	Scab	Erosivity Factor in Cold Particle Erosion Test		Hot Erosion/Oxidation
							s/m x 10^4	(sec/mil)	
8HA2	SOA 1976 + 3 g BN	HP SiC	71	87	(4)	Layer fractured	7.33	(1.86)	Slight erosion of layer at edges
9HB1(2)	SOA 1976	Silcomp	60(3)	78	0.400	Yes, slight pullout	7.37	(1.87)	Slight layer erosion at edges
9HB2	SOA 1976	Silcomp	58	80	0.266	Yes	6.78	(1.72)	Slight layer erosion at edges
11HA1	SOA 1976 + 3 g BN	HP SiC	74	89	0.333	Yes, layer pullout	7.09	(1.80)	Slight layer erosion at edges
11HA2	SOA 1976 + 3 g BN	HP SiC	76(3)	94	0.444	Yes, layer pullout	7.09	(1.80)	Slight layer erosion after seven cycles at 1589 K (2400° F)
11HB(2)	SOA 1976 + 3 g BN	HP SiC	76	ND	ND	ND	---	ND	Missing after 15 cycles at 1589 K (2400° F)

(1) Average of 9 readings

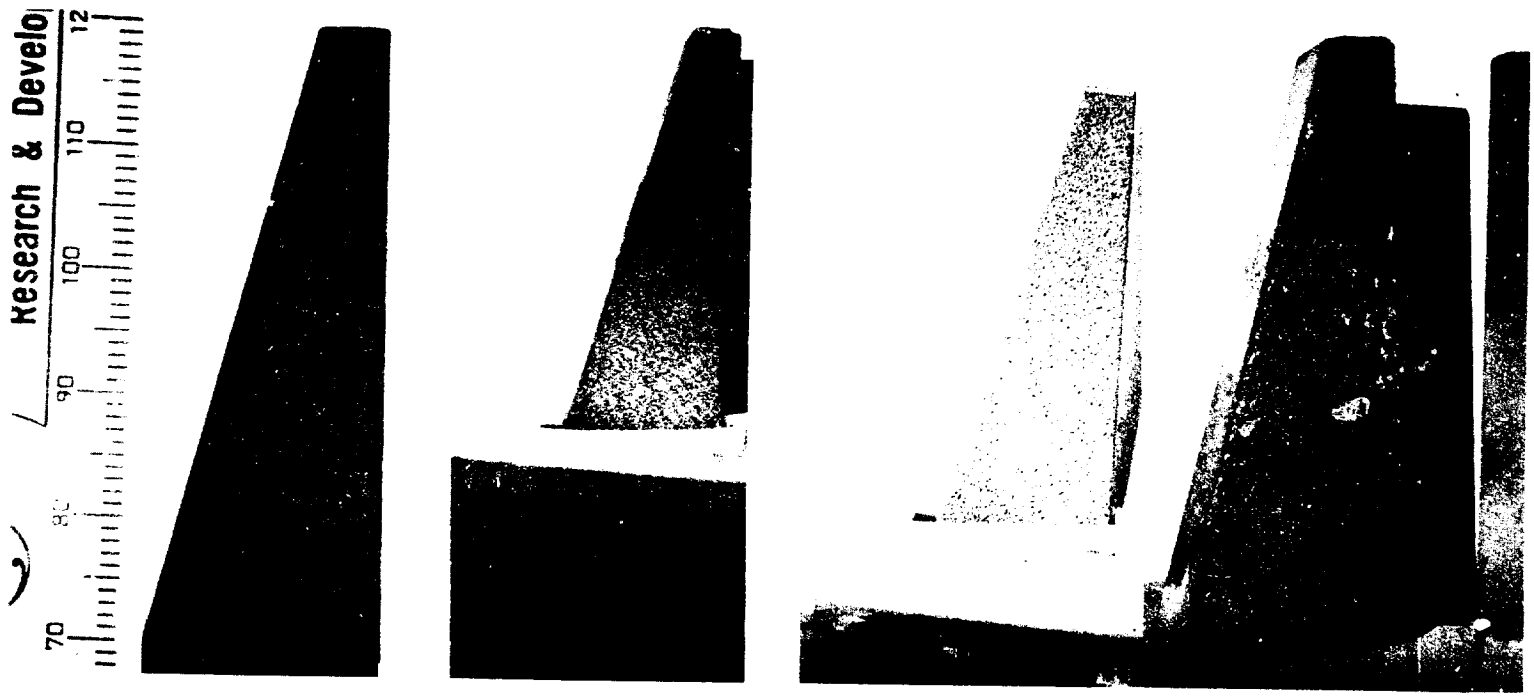
(2) Sample preoxidized at 1366 K (2000° F) for 65 hrs in static air.

(3) Preoxidation hardness

(4) Test stopped, layer fractured.

ND = No posttest data, specimen came out of the holder and is missing.

HP = Hot pressed



(a) As Fabricated

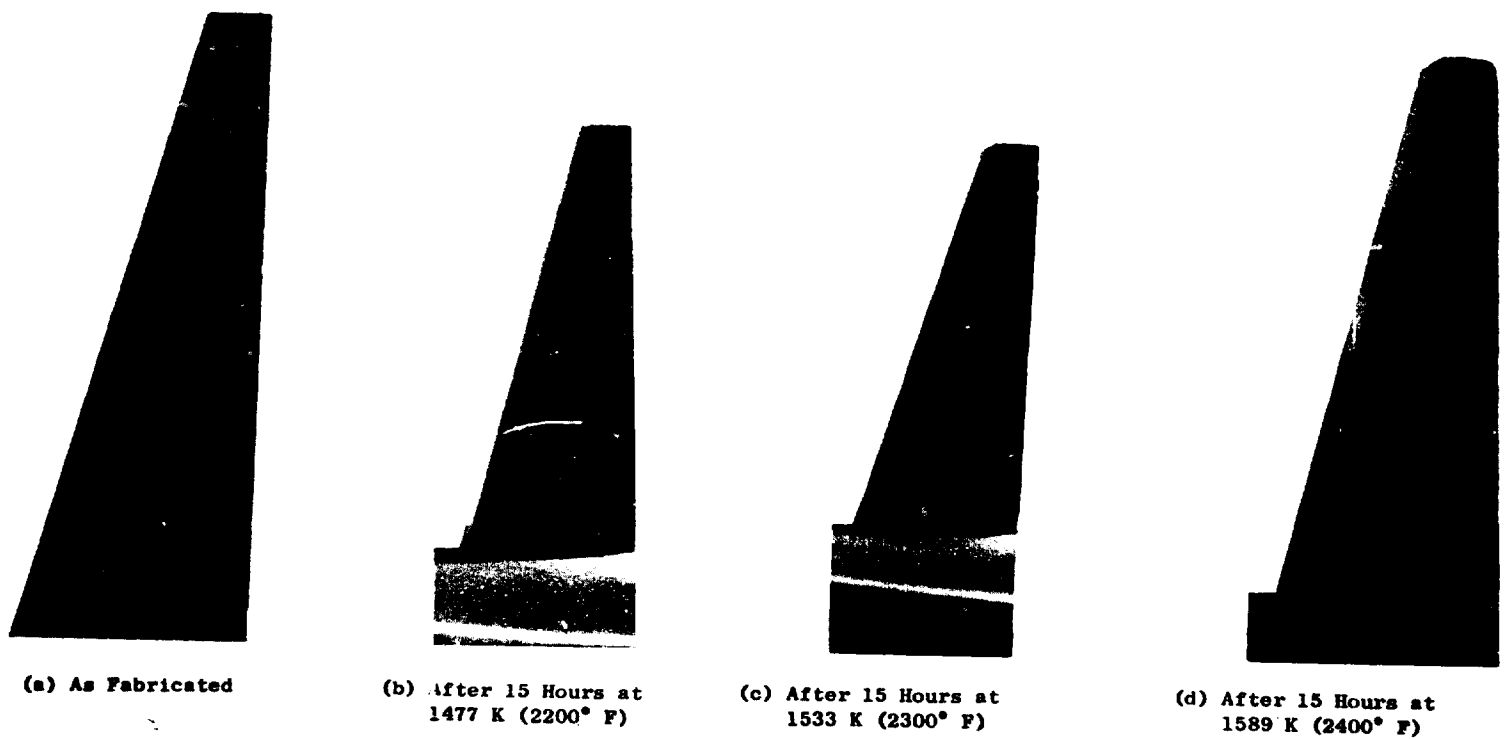
(b) After 15 Hours at 1477 K (2200° F)

(c) After 15 Hours at 1533 K (2300° F)

(d) After 15 Hours at 1589 K (2400° F)

Figure 54. Third Task III Mach 1.0 Gas Oxidation/Erosion Test Specimens 8HA2.

Figure 55. Third Task III Mach 1.0 Gas Oxidation/Erosion Test Specimens 11HB2.



(a) As Fabricated

(b) After 15 Hours at 1477 K (2200° F)

(c) After 15 Hours at 1533 K (2300° F)

(d) After 15 Hours at 1589 K (2400° F)

Figure 56. Third Task III Mach 1.0 Gas Oxidation/Erosion Test Specimen 9HB2.

Figure 57. Typical Microstructure of a Specimen After the Third Task III Mach 1.0 Gas Oxidation/Erosion Test at 1473/1533/1589 K (2200°/2300°/2400° F) Showing SiO_2 Film Present Around Some of the Si/SiC Network.

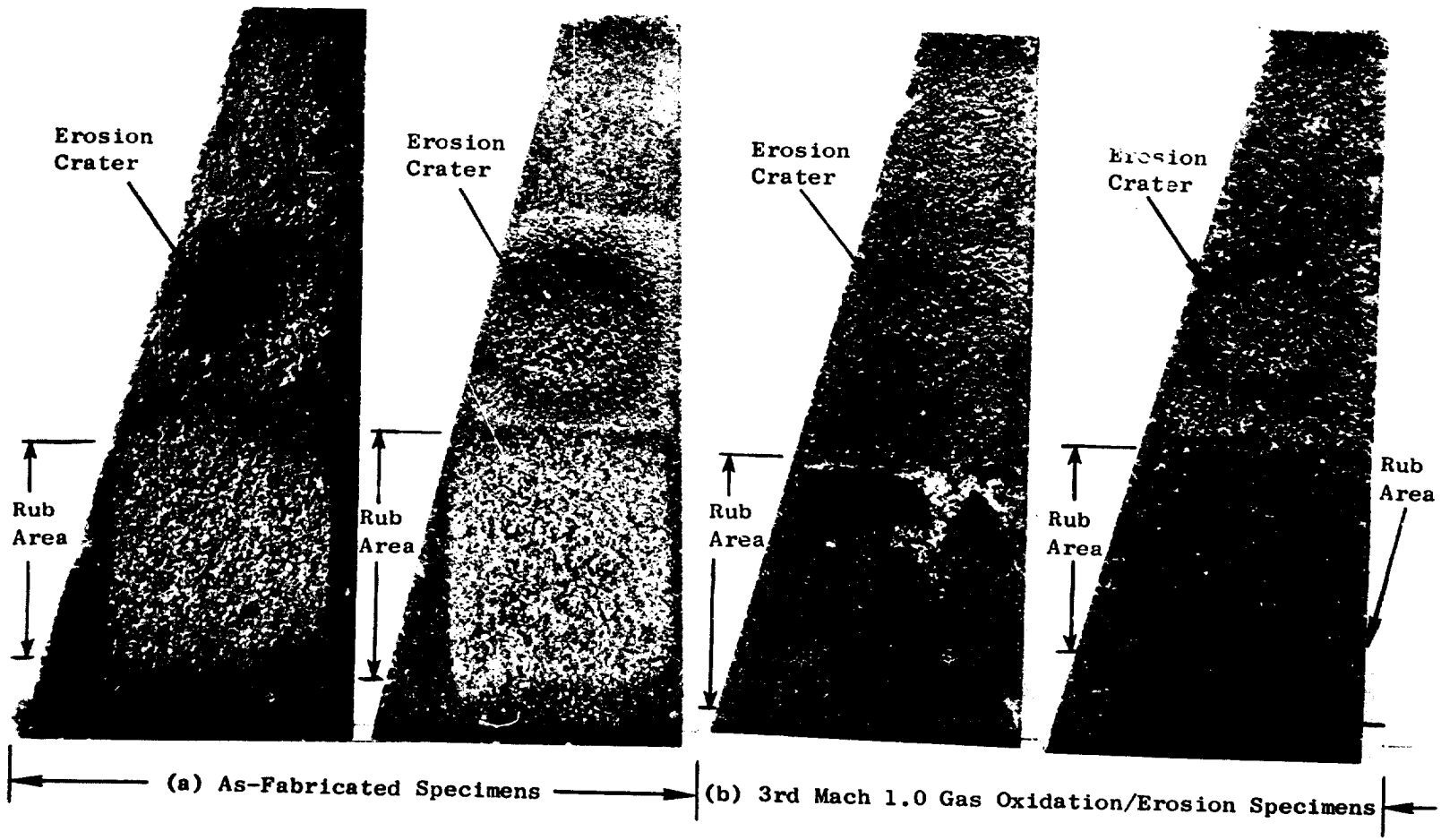


Figure 58. Cold Particle Erosion and Room Temperature Rub Test Results on As-Fabricated Specimens and Third Mach 1.0 Gas Oxidation/Erosion Specimens.

than 75 have a tendency for blade material transfer. The posttest cold particle erosion resistance did not change significantly. The Task III Mach 1.0 gas oxidation/erosion test and posttest evaluations indicated that although the hot gas erosion resistance of the BN-containing layers was improved by preoxidation, the abradability of such layers was not satisfactory.

The following conclusions were drawn from the Mach 1.0 gas oxidation/erosion tests conducted in this program:

1. The as-fabricated layers with surface hardnesses (R_{15Y}) below 50 eroded excessively, and layers with hardnesses above 50 showed acceptable hot gas erosion resistance at least up to 1477 K (2200° F). At temperatures above 1477 K (2200° F), the specimens in the as-fabricated condition eroded excessively. Improvements in the hot gas erosion behavior at temperatures above 1477 K (2200° F) were obtained by preoxidizing the specimens prior to the test or in the test at 1477 K (2200° F).

2. The preoxidized layer specimens showed improved hot gas erosion resistance. However, the surface hardness of the layers increased due to preoxidation exposure, consequently increasing blade wear in the rub tests. The abradability of these layers was not satisfactory. The satisfactory combination of hot gas erosion resistance and abradability was not obtained.

3. The formation and retention of silicon dioxide by oxidation of free silicon in the Si/SiC network affected hot gas erosion resistance and abradability of the BN-containing rub layers. The oxide retention is necessary for adequate hot gas erosion resistance. The limited data generated in this program indicated that the oxide is formed as a continuous film around the Si/SiC network and can be retained up to 1477 K (2200° F) in the Mach 1.0 gas erosion environment. Above 1477 K (2200° F) the oxide tends to be viscous and becomes discontinuous around the network and, therefore, may not provide adequate long-term protection.

4. There was a large data scatter primarily because the process for fabricating the rub layers is not yet fully reproducible. BN as an additive was found to be the main variable controlling the microstructure and properties of the rub layers. The effects of other variables such as BN particle size and distribution, comminution of the BN particles during preparation of layers, and the furnace cycle (temperature, time and heating rate) need to be studied in order to increase the reproducibility of the fabrication process.

5.0 CONCLUSIONS AND RECOMMENDATIONS

1. The silicon carbide composite structures with attached BN-containing Si/SiC rub layers have shown good abradability and resistance to oxidation, gas erosion, thermal shock, and ballistic impact damage up to 1477 K (2200° F).

2. For 1644 K (2500° F) application, a satisfactory combination of hot gas erosion resistance and abradability was not achieved. Further improvement in hot gas erosion resistance may be achieved by: (1) varying the relative amounts of SiC, BN, and free silicon; (2) a preoxidizing treatment; (3) a study of the role of free silicon, specifically its formation and retention characteristsics and methods to retain the silicon dioxide which is formed during oxidation exposure.

   Improved hot gas erosion resistance may also be achieved by using harder layers containing low BN additives. Such layers will be less abradable and will require the use of abrasive tipped blades. Any future ceramic turbine seal development program should include efforts to develop an abrasive tip system which can rub into the ceramic seal without causing excessive blade wear.

3. The BN-free Si/SiC layers showed better abradability, cold particle erosion resistance, oxidation resistance, and ballistic impact resistance than BN-containing Si/SiC rub layers. A technique to bond these layers to a dense ceramic block is needed for further evaluation. A bonding technique utilizing pure silicon was studied but needs further improvements in bond strength and reproducibility.

4. Both BN-free and BN-containing Si/SiC rub layers showed marked improvements over the baseline material, Bradelloy 500, in oxidation resistance, erosion resistance, abradability, and ballistic impact resistance.

5. Although the ceramic seal materials have shown marked improvement over Bradelloy 500, a successful application of ceramic materials will require additional work in the following areas:

   - Attachment of ceramic components to the nickel- and cobalt-based superalloy components
   - Reliable data base for safe design and performance
   - New design concepts incorporating the brittle nature of ceramic materials
   - Improved quality control, nondestructive evaluation, and life-prediction methods
   - Reproducible and near net shape processing techniques.

6.0 REFERENCES

1. Bessen, I.I., Rigney, D.V., and Schwab, R.C., "Improved High Pressure Turbine Shroud, Final Report," NASA Contract NAS3-18905, NASA CR-135181, February 1977.