THE AMERICAN SOCIETY OF MECHANICAL ENGINEERS
Three Park Avenue, New York, N.Y. 10016-5990

99-GT-190

BREAK

ADVANCES IN OXIDE-OXIDE CMC

Dr. Robert A. Jurf, Mr. Steven C. Butner
Composite Optics, Inc.
9617 Distribution Ave.
San Diego, CA 92121

ABSTRACT

Recent advances in COI's oxide-oxide CMC materials will be presented including basic processing steps, updated material properties, and fabrication techniques. Material properties of COI's alumino-silicate system reinforced with various oxide fabrics will be compared, along with progress in developing a 1200°C oxide matrix system for future turbine system applications. Examples of fabricated hardware, including a subscale combustion liner, will be shown. Recent test and evaluation data will be provided.

INTRODUCTION

During the past three years, Composite Optics, Inc. (COI) has developed oxide-oxide ceramic matrix composites (CMCs) for advanced military aircraft, with potential applications in commercial gas turbine engine systems. Extreme temperature conditions are realized for very brief periods of time for missile applications (less than one hour) and up to 100 hours for a military engine in afterburner conditions. These temperatures are relatively short compared to the 10,000's of hours of steady state operation in commercial gas turbines and other industrial applications.[1] COI has explored the viability of using an oxide system in commercial energy applications by characterizing coupons in ways directed toward commercial requirements. The baseline alumino-silicate system has shown early promise in both basic properties, producibility, and environmental resistance. As COI proceeds with testing and evaluation of subscale rig components, a more refractory oxide system aimed at satisfying the higher temperatures and longer durations of the gas turbine engine community is being developed.

CMC DEVELOPMENT OBJECTIVES AND APPROACH

The original development goals were established for current and future military exhaust systems. In addition to meeting aircraft thermomechanical demands, CMC systems must have a simple robust process in order to be economically viable. Specific targets were identified at the onset of development:

(1) tensile strength of 140 MPa at 1000°C with 1100°C excursions
(2) mechanical property stability with temperature
(3) low cost manufacturing process

Presented at the International Gas Turbine & Aeroengine Congress & Exhibition
Indianapolis, Indiana — June 7–June 10, 1999
This paper has been accepted for publication in the Transactions of the ASME
Discussion of it will be accepted at ASME Headquarters until September 30, 1999

COI's ceramic matrix composite baseline approach is a sol-gel derived alumino-silicate matrix that can be combined with a variety of commercially available fiber reinforcements such as Nextel 550, Nextel 610, and Nextel 720.[2] The baseline oxide-oxide system relies on controlled matrix porosity for toughness, thereby eliminating the need for fiber coatings. Recent findings support the conclusion that high strength, damage tolerant oxide-oxide CMC's can be made without the use of fiber coatings. [3,4,5]

Reinforcement Selection

The fibers used are all in fabric form and are selected based on the advantages that each type offers when laminated in COI's ceramic composite (Table 1). The maximum use temperature of COI's CMCs is largely determined by the fabric reinforcement system. Of the oxide fibers currently available in the commercial market, Nextel 720 has the best temperature capability and creep resistance.

Table 1. Features of Different CMC Fabric Reinforcements

	Oxide CMC Properties with Various Oxide Fabrics				
	Astroquartz	**Nextel 312**	**Nextel 550**	**Nextel 720**	**Nextel 610**
Composite Density (gm/cc)	1.50	2.30	2.41	2.60	2.83
Nominal Fiber Volume (%)	50	48	38	48	51
RT Tensile Modulus (GPa)	22 (est)	31.0	40.0	76.5	124.1
RT Tensile Strength (MPa)	46 (est)	124.8	147.6	179.0	366.1
CTE (ppm/C)	0.40	4.85	5.40	6.30	7.92

Typical Properties of Nextel 720 CMC

Mechanical testing has been an ongoing part of the maturation process for this CMC system. Table 2 summarizes basic mechanical strength and modulus values compiled to date on the Nextel 720 alumino-silicate system. All tests used typical ASTM standards for organic composites and/or ceramic matrix composites.

Table 2. Typical Properties of Nextel 720 Reinforced Alumino-Silicate CMC System

	Temp	Strength (MPa)	Modulus (GPa)	Strain (%)
Tension (17)	RT	179	76.5	0.30
Tension (after 100 hours at 1000°C) (6)	RT	187	82.1	0.29
Tension (4)	1000°C	170	73.1	0.27
Tension (after 100 hours at 1000°C) (4)	1000°C	176	78.6	0.28
Flexure (48)	RT	216	97.9	0.22
Compression (12)	RT	186	80.0	0.22
In-Plane Shear (12)	RT	31	14	0.49
Interlaminar Shear Strength (45)	RT	11.5	-	-

(*) Quantities of test specimens

Elevated Tensile Data[6]

Creep and Fatigue Properties

Resistance to creep rupture and fatigue is an important material characteristic for both military and commercial gas turbine engines. Larry Zawada at the Air Force Research Laboratory (AFRL) Materials and Manufacturing Directorate, Wright-Patterson AFB, Ohio, has characterized COI's Nextel 720 alumino-silicate system at 1000°C and 1100°C.[7] Preliminary results summarized in Table 3 are very encouraging. The residual strengths after fatigue are equal or greater than the virgin untested material, suggesting that repeated loading may have a work-hardening toughening phenomenon. The creep rupture tests are just as encouraging, with residual strengths after 100 hours at 150 MPa equal to 209 MPa. Tests are continuing, and data will be reported as it becomes available.

Table 3. Residual Tensile Strengths after Fatigue and Creep Loadings in Air

	Test Temperature	Stress Level	Loading Conditions	Residual Tensile Strength
Fatigue	1000°C	125 MPa	100,000 cycles	193 MPa
	1000	150	100,000	211
	1000	160	100,000	195
	1100	150	100,000	168
Creep Rupture	1000	150	100 hours	209

Environmental Resistance

Rob Kowalik at NAVAIR Materials Division at Patuxent River, Maryland completed preliminary hot corrosion studies in March 1998 of COI's Nextel 550 and 720 reinforced CMC.[8] The first test run on the materials was a 500 cycle oxidation resistance test to 900°C in air only; each cycle consisting of 55 minutes at 900°C and 5 minutes at room temperature. As expected, the CMC was extremely inert to the oxidation exposure; there was no weight change or visible change in the CMC samples.

The second test run on the material was a hot corrosion thermal exposure. When salt water is ingested into aircraft turbine engines and mixed with sulfur residue, corrosive salts are deposited on nozzle parts, greatly reducing their useful operating life. To model this behavior, Na_2SO_4 is deposited on the CMC coupon surfaces at 5 gm/ cm^2. The 500 cycle thermal exposure to 900°C is repeated. In materials such as SiC/SiC, a thick film of sodium silicate forms on the surface which spalls off during continued thermal cycling.[9] After completing 500 cycles of cycling, no visible glass formation and no significant weight changes were reported. Initial analysis gave no indication of any sodium silicate layer formation. The surface topography and morphology looked unchanged. The results showed COI's CMC materials were well-behaved at 900°C and are very promising. Additional evaluation is being pursued by the Navy, National Aeronautics and Space Administration (NASA), and Oak Ridge National Laboratory (ORNL) at this time, and will be reported in future publications.

Effects of Long-Term Aging

Siemens-Westinghouse has carried out long-term aging studies on COI's Nextel 720 alumino-silicate system. Samples were exposed up to 1000 hours at temperatures ranging from 1000°C to 1200°C (Figure 1) in air.[10] The data indicates that the properties of the Nextel 720 composite are stable for very long-term exposures (1000 hours) up to 1000°C, then degrade rapidly beginning at 1100°C. COI has carried out similar aging studies for durations up to 100 hours and temperatures up to 1200°C. While the results were very similar to the Siemens-Westinghouse data at 1200°C, the results at 1100°C differed substantially. COI's results indicated less than a 10% degradation in strength after 100 hour exposure to 1100°C. Siemens-Westinghouse found over a 40% degradation in strength under the same conditions. While the details of the results differ, the overall conclusions are the same.

Along with the degradation in strength with temperature exposure beyond 1100°C, there is a transition from fibrous to brittle failure (Figure 2). While the mechanism for the strength/microstructural degradation has not been fully characterized, COI currently believes that the primary loss in strength is a result of additional matrix densification rather than fiber degradation. As the matrix is further densified (through sintering) it shrinks, becomes stronger, and bonds more extensively to the reinforcement fibers thereby degrading the toughness mechanism.

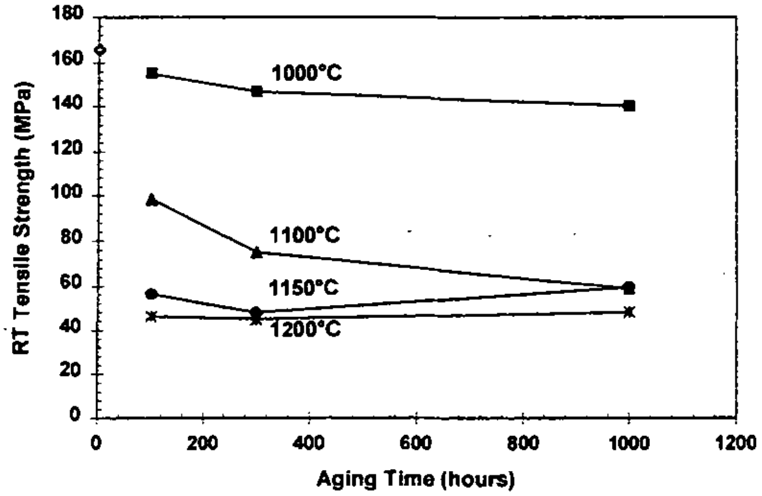

Figure 1. Tensile Strength Retention after 1000 Hours of Exposure up to 1200°C[10]

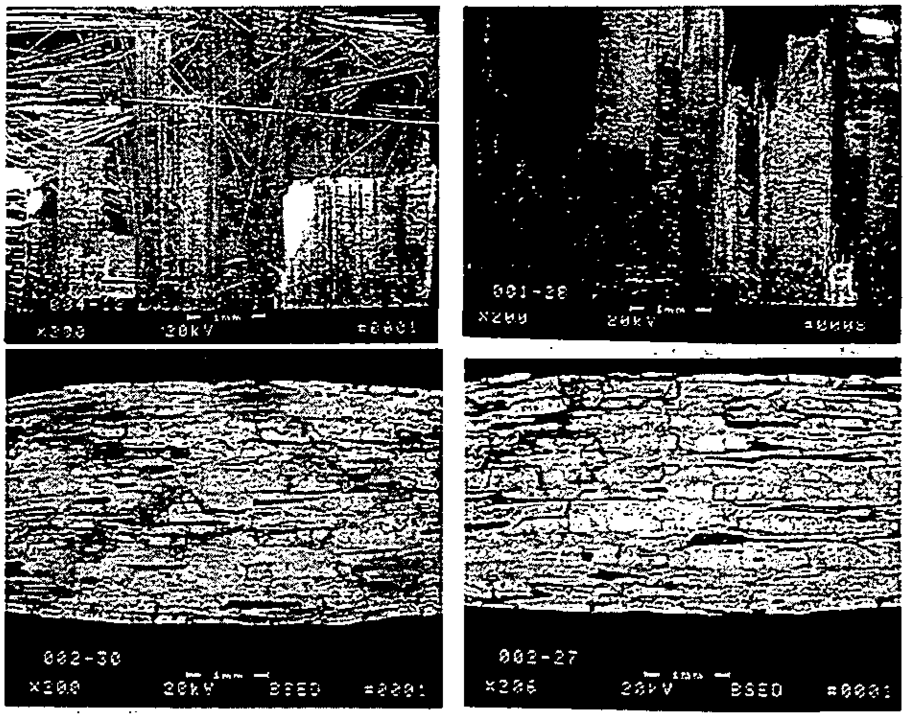

Figure 2. Fracture Surfaces as Fabricated (top-left), and after 1000 Hour Exposure to 1000°C (top right), 1100°C (bottom left) and 1200°C (bottom right).[10]

The data strongly suggests that COI's alumino-silicate Nextel 720 system will not satisfy the 10,000 hour 1200°C demands of next generation gas turbine engines. The Future Work section presents basic research COI has initiated on more refractory oxide systems that are aimed at commercial gas turbine engine applications.

COMPONENT DESIGN AND MANUFACTURING

COI's process for manufacturing oxide-oxide CMCs is simple and low cost. The fabrication process does not require repetitive re-infiltration or pyrolyzation steps. No thin fiber coatings or exterior oxidation protection coatings are required for COI's baseline alumino-silicate process. This lowers the fabrication costs and eliminates coating compatibility and thermal stability problems. Even "low cost" CMCs can be prohibitively expensive due to tooling and complex stiffener arrangements. A conventional approach to fabricating a complex shaped component is shown in Figure 3.

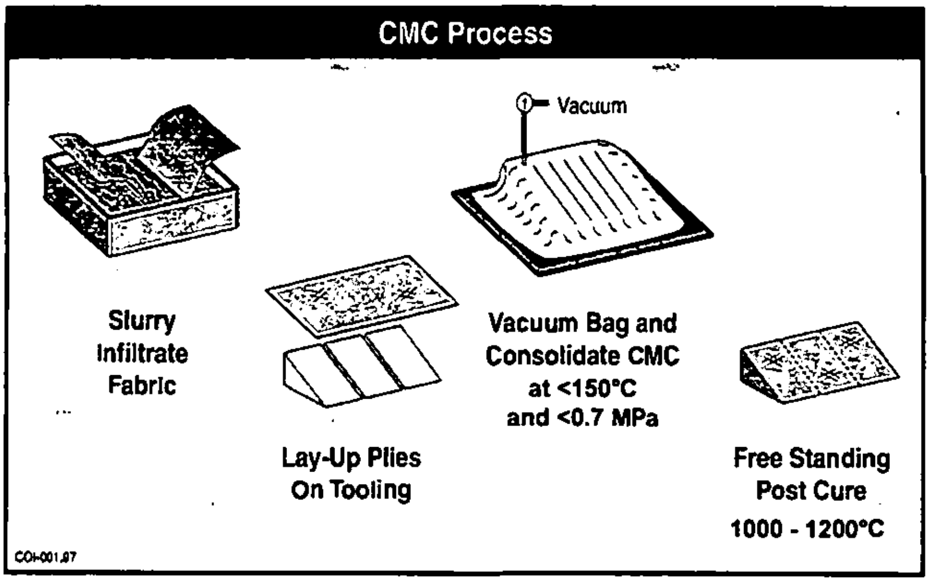

Figure 3. COI's CMC Fabrication Process

The matrix is a viscous slurry that is prepregged into the fabric and staged to a tacky consistency. Components are laid-up using organic composite techniques and cured (dried) using low pressures (<0.7 MPa) and temperatures (<150°C). Composite parts have sufficient strength at this point to be demolded. The final step required is a free standing pressureless sinter of about 1150°C to sinter the matrix to final density listed in Table 1.

We applied this process to a subscale liner to be tested by Solar Turbines in San Diego, California. This liner test has been used by Solar to screen various CFCC's for suitability in gas turbine engines.[1] The liner COI produced was nominally 20 cm in diameter and 20 cm in length. The wall architecture was six plies, and about 1.65 mm thick. The manufacturing sequence is shown in the sequence of photos, Figure 4.

With the tooling in-hand, the time required to prepare the material, lay-up and cure the composite, and trim the ends was six working days. In a production environment, this could be reduced even further, making this type of manufacturing process economically attractive.

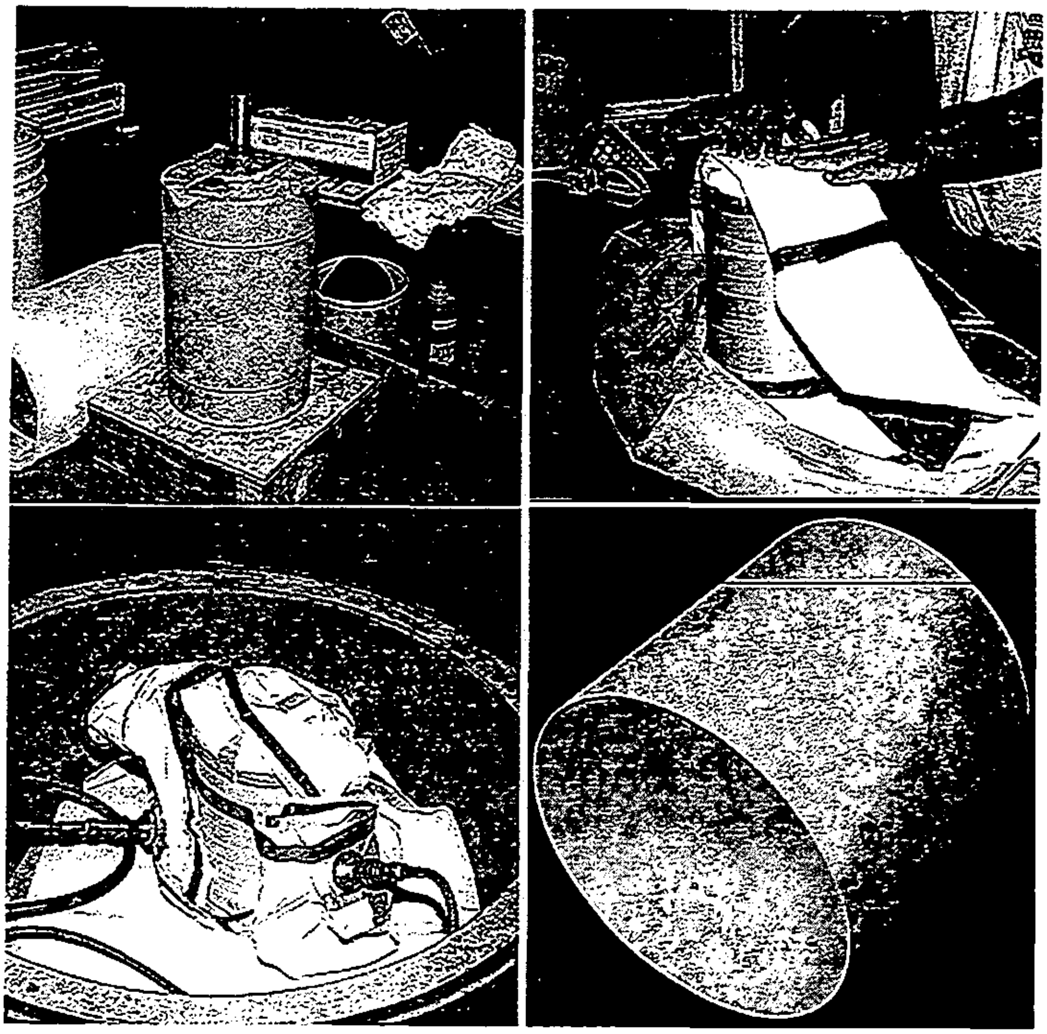

Figure 4. The Tooling (Top-Left), Ply Lay-Up (Top-Right), Bagging Process (Bottom Left) And Finished Part (Bottom Right)

FUTURE WORK

In May of 1998, COI was awarded a Phase I Small Business Innovative Research (SBIR) contract by AFRL to investigate the development of a low cost oxide matrix that is stable for long term exposures to 1200°C. The primary objective is to develop and demonstrate an oxide matrix composite using commercially available refractory fibers (Nextel 720) that will retain better than 90% of its original composite strength after 100 hours of exposure to 1200°C. The mechanical property goals were to achieve a 175 MPa tensile strength, 200 MPa flexural strength and a 12 MPa interlaminar shear strength in a zero degree warp aligned fabric layup. Several oxide matrices were investigated including an alumina and mullite based system. Preliminary Phase I results have shown promise in the approach. Some early laminate test results are in Table 4.

Table 4. Phase I SBIR Results Using Nextel 720 Fabric

	Flexure Strength (MPa)		
Matrix Composition	As Cured	100 hours at 1200C	% Strength Retention
Baseline System	208.6	88.3	42%
Alumina	182.0	190.3	105%
Mullite	124.8	115.1	92%

All of the laminates produced in the first iteration achieved better than 90 % strength retention after 100 hour exposure to 1200°C. In Table 4 the strength retention of COI's baseline Nextel 720 system is shown for comparison. The alumina based system has shown better initial structural performance while the mullite system showed better microstructural development. Both systems showed very good strength retention properties. Future work will focus on improving the strength and more extensively characterizing the temperature stability of the materials. A follow-on to the SBIR Phase I program has been awarded by AFRL. The Phase II effort will involve research activities at AFRL, Siemens-Westinghouse, Solar Turbines and ORNL.

ACKNOWLEDGEMENTS

We gratefully acknowledge the support of the Air Force Research Laboratory under SBIR Contract F33615-98-C-5210, and we wish to thank Larry Zawada of AFRL for performing fatigue and creep rupture tests and for providing these data. We also acknowledge Jay Lane and Eric Carelli, Siemens Westinghouse Power Corporation in Pittsburgh, PA for the long-term aging and photomicrographs. Lastly, we acknowledge Solar Turbines for funding to manufacture the subscale liner.

REFERENCES

[1] Simpson, J. et al, 1997, "Testing of Monolithic Ceramics and Fiber Reinforced Ceramic Composites for Gas Turbine Combustors," Ceramic Engineering and Science Proceedings, Vol. 18, Issue 4.

[2] 3M Ceramic Fiber Technical Data Brochure, 1998.

[3] Tu, W.C. et al, 1996, J. Am. Ceram., "Concept for a Damage-Tolerant Ceramic Composite with "Strong" Interfaces," Soc., 79 [2] 417-24.

[4] Cazzato A. et al, 1997, "Monazite Interface Coatings in Polymer and Sol-Gel Derived Ceramic Matrix Composites", presented at the 21st Annual Cocoa Beach Conference.

[5] Levi ,C., 1997, "Damage Tolerant All-Oxide CMC's Without Fiber Coatings," presented at the 21st Annual Cocoa Beach Conference.

[6] Personal Communication with J. French, Boeing, St. Louis, MO.

[7] Personal Communication with L. Zawada, AFRL, Dayton, OH.

[8] Personal Communication with R. Kowalik, NAVAIR, Patuxent River, MD.

[9] Wang, S.W. et al, 1994, "High Temperature Behavior of Salt Coated Nicalon Fiber Reinforced Calcium Alumino-silicate Composite," Ceram. Eng. Sci. Proc. Vol. 15, No. 4, pp. 465-474.

[10] Personal Communication with J. Lane and E. Carelli, Siemens Westinghouse, Pittsburgh, PA.